Supplementary data for the article:

Pavlović, R. Z.; Bjelaković, M. S.; Milić, D. Diamide-Based Fullerosteroidal and Disteroidal [2] Rotaxanes: Solvent-Induced Macrocycle Translocation and/or Unthreading. *RSC Advances* **2016**, *6* (43), 37246–37253. https://doi.org/10.1039/c6ra03872g

Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary Information

Diamide-based fullerosteroidal and disteroidal [2]rotaxanes: solvent-induced macrocycle translocation and/or unthreading

Radoslav Z. Pavlović, Mira S. Bjelaković and Dragana R. Milić*

Contents

Table S1. ^{1}H NMR chemical shifts of selected signals of thread **3a** and rotaxane **4a** obtained in $CDCl_3$ and $[D_6]DMSO$

Signal	Thread **3a** δ (ppm)	Rotaxane **4a** δ (ppm)	$\Delta\delta$ (ppm)	Thread **3a** δ (ppm)	Rotaxane **4a** δ (ppm)	$\Delta\delta$ (ppm)
	$CDCl_3$			$[D_6]DMSO$[b]		
NH(Gly)	6.92	5.90	1.02	8.11	7.96	0.15
NH(GABA)	6.67	6.85	0.18[a]	7.83	7.96	0.13[a]
CH_2(Pyrr)	4.43	4.27	0.16	4.45	3.38	1.07
CH_2(Gly)	3.97	2.83	1.14	3.78	3.66	0.12
CH_2(4')-GABA	3.30	3.06	0.24	3.16	2.92	0.24
CH_2(4")-GABA	3.17	2.94	0.24	3.13	2.05	1.08
CH_2(3')-GABA	1.82	1.66	0.16	1.72	1.48	0.24
CH_2(3")-GABA	2.29	1.91	0.38	2.19	1.45	0.74
CH_2(2')-GABA	2.34	2.17	0.17	2.29	2.03	0.26
CH_2(2")-GABA	2.67	2.25	0.42	2.54	1.88	0.66

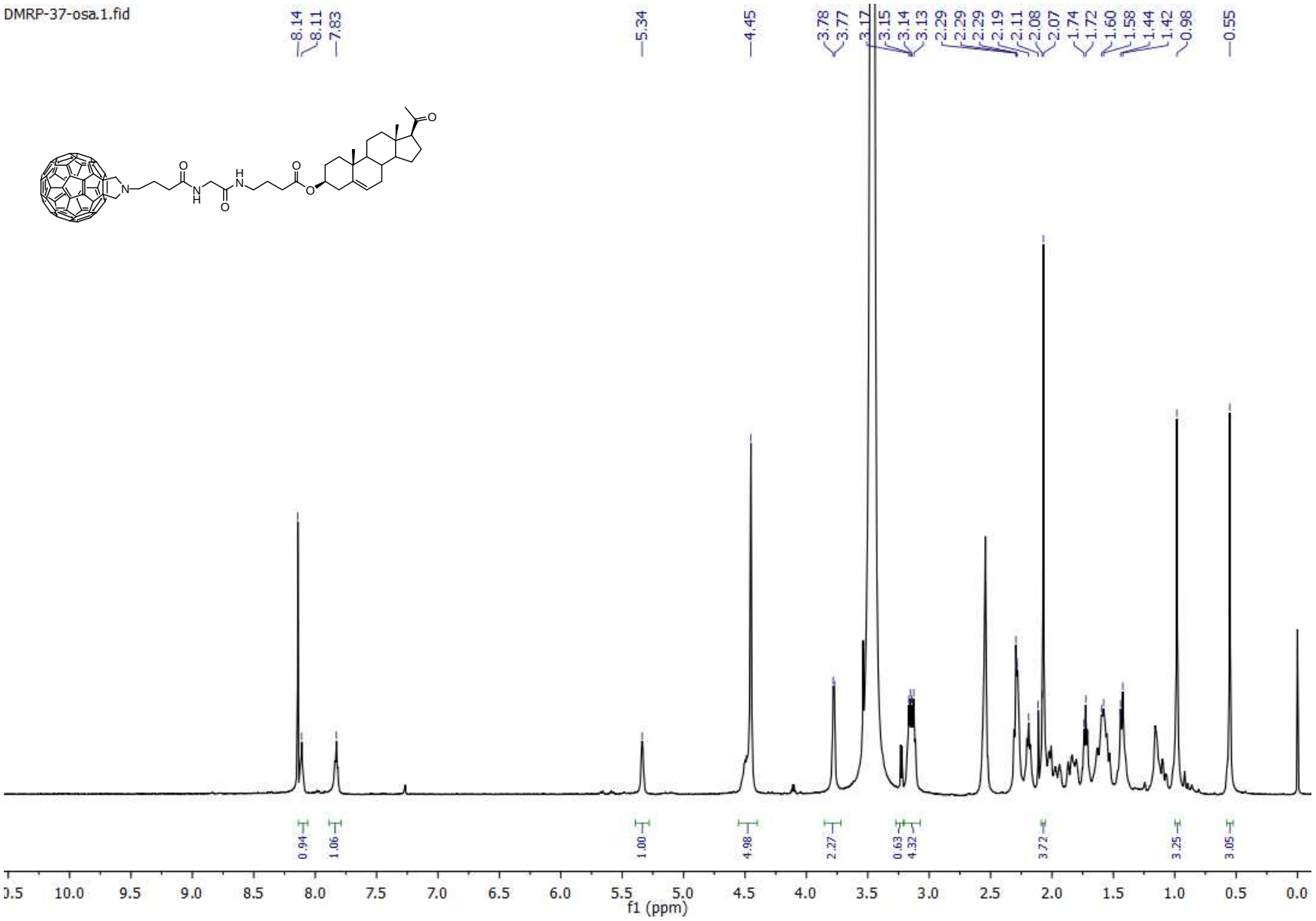
[a] $\Delta\delta$ is negative (thread-rotaxane); absolute values are showed; [b]Spectrum was obtained with addition of a one drop of $CDCl_3$ because of insolubility of rotaxane in $[D_6]DMSO$

Table S2. ^{1}H NMR chemical shifts of selected signals of thread **3b** and rotaxane **4b** in $CDCl_3$ and $[D_6]$DMSO

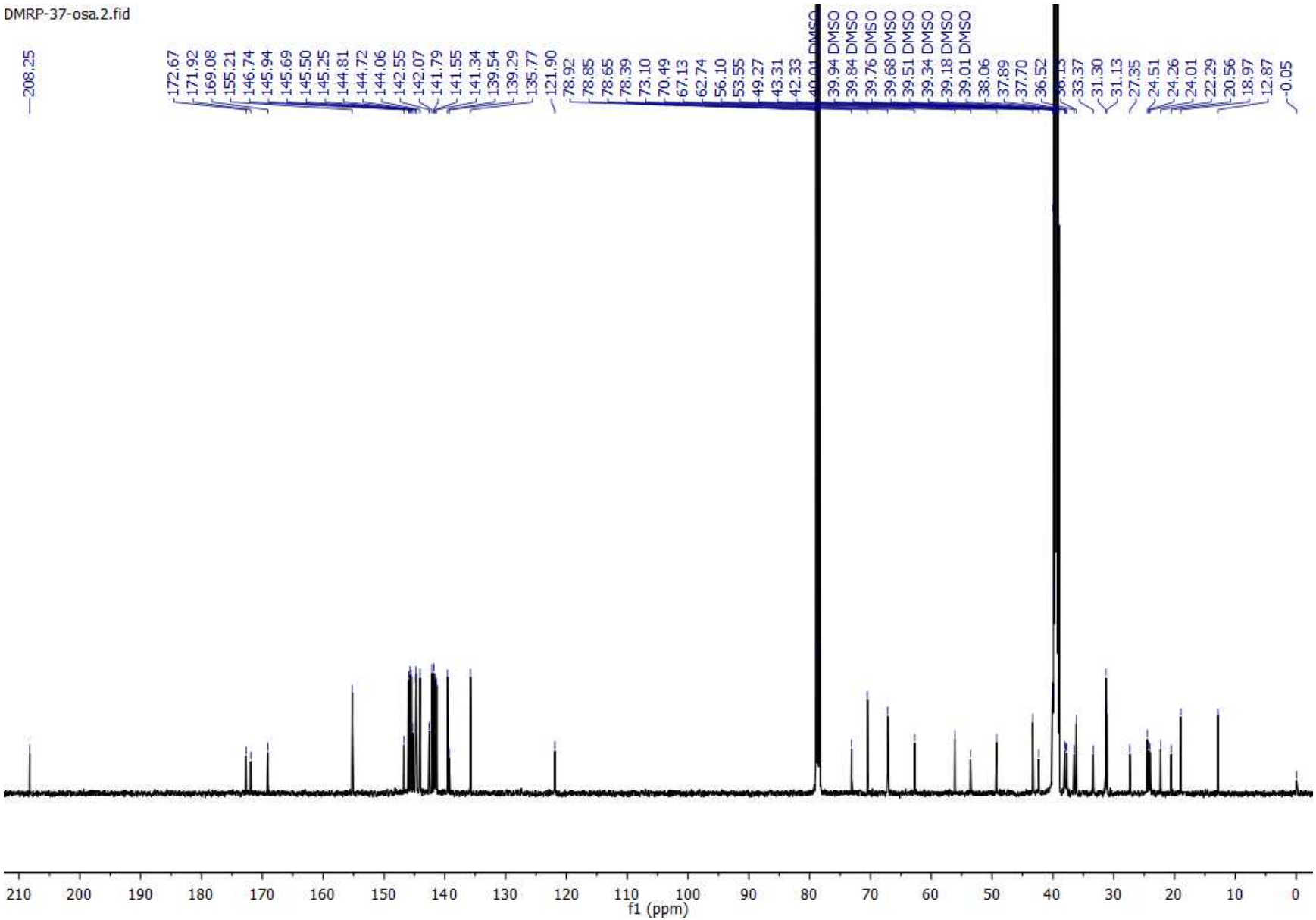
Signal	Thread **3b** δ (ppm)	Rotaxane **4b** δ (ppm)	$\Delta\delta$ (ppm)	Thread **3b** δ (ppm)	Rotaxane **4b** δ (ppm)	$\Delta\delta$ (ppm)
	$CDCl_3$			$[D_6]$DMSO		
NH(Gly)	6.47	5.95	0.52	8.16	7.86	0.30
NH(GABA)	6.79	7.10	0.31[a]	7.68	7.64	0.04
CH_2(Gly)	3.93	2.87	1.06	3.61	3.38	0.23
CH_2(4)-GABA	3.29	3.06	0.23	3.06	2.58	0.48
CH_2(3)-GABA	1.84	1.66	0.18	1.63	1.18	0.45
CH_2(2)-GABA	2.33	2.14	0.19	2.25	1.73	0.52
CH_2(3)-Succ	2.70	2.32	0.38	2.49	2.10	0.39
CH_2(2)-Succ	2.49	2.09	0.40	2.39	2.10	0.29

[a] $\Delta\delta$ is negative (thread-rotaxane); absolute values are showed

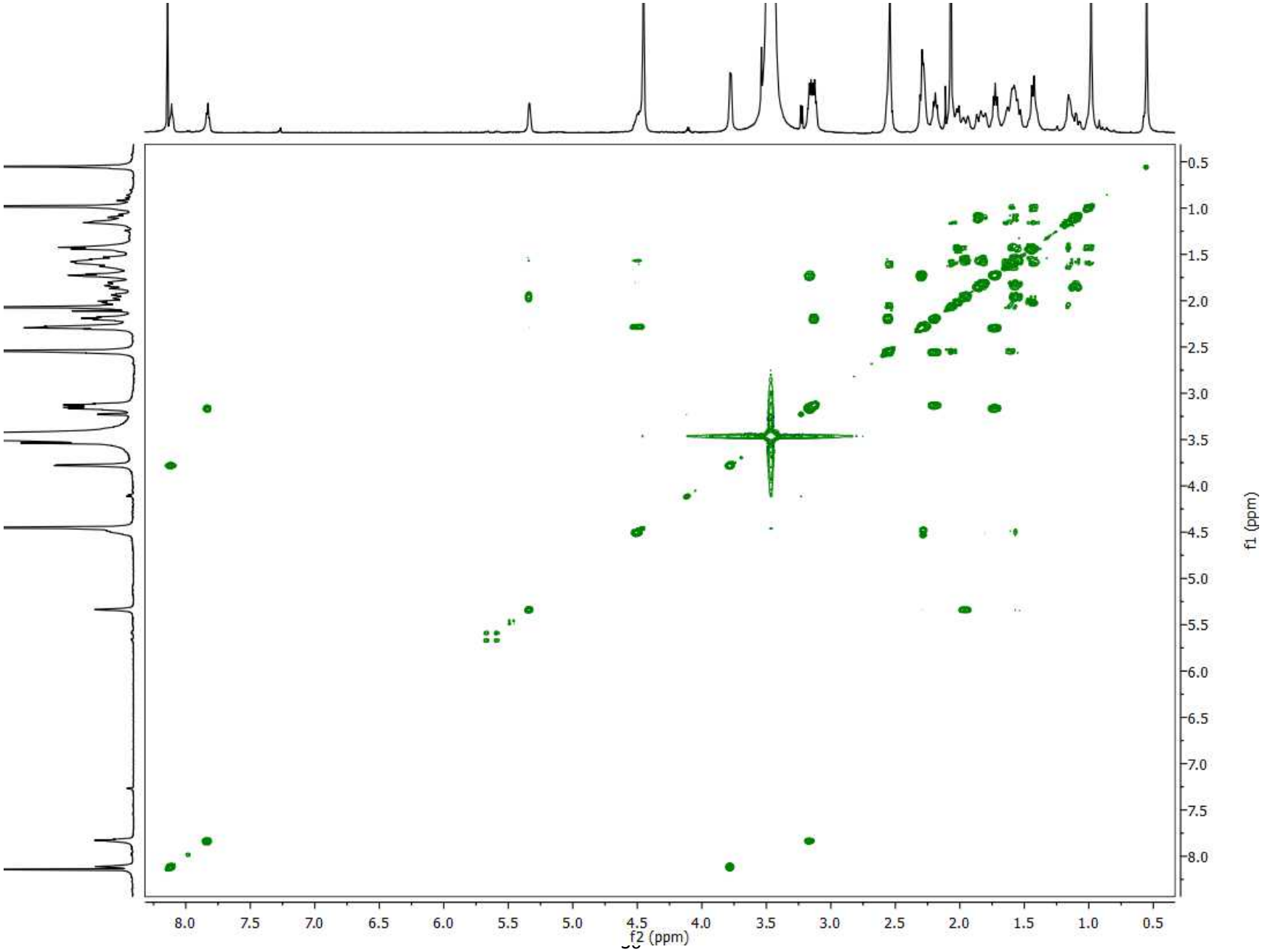
^{1}H NMR spectrum of thread **3a** in [D_6]DMSO



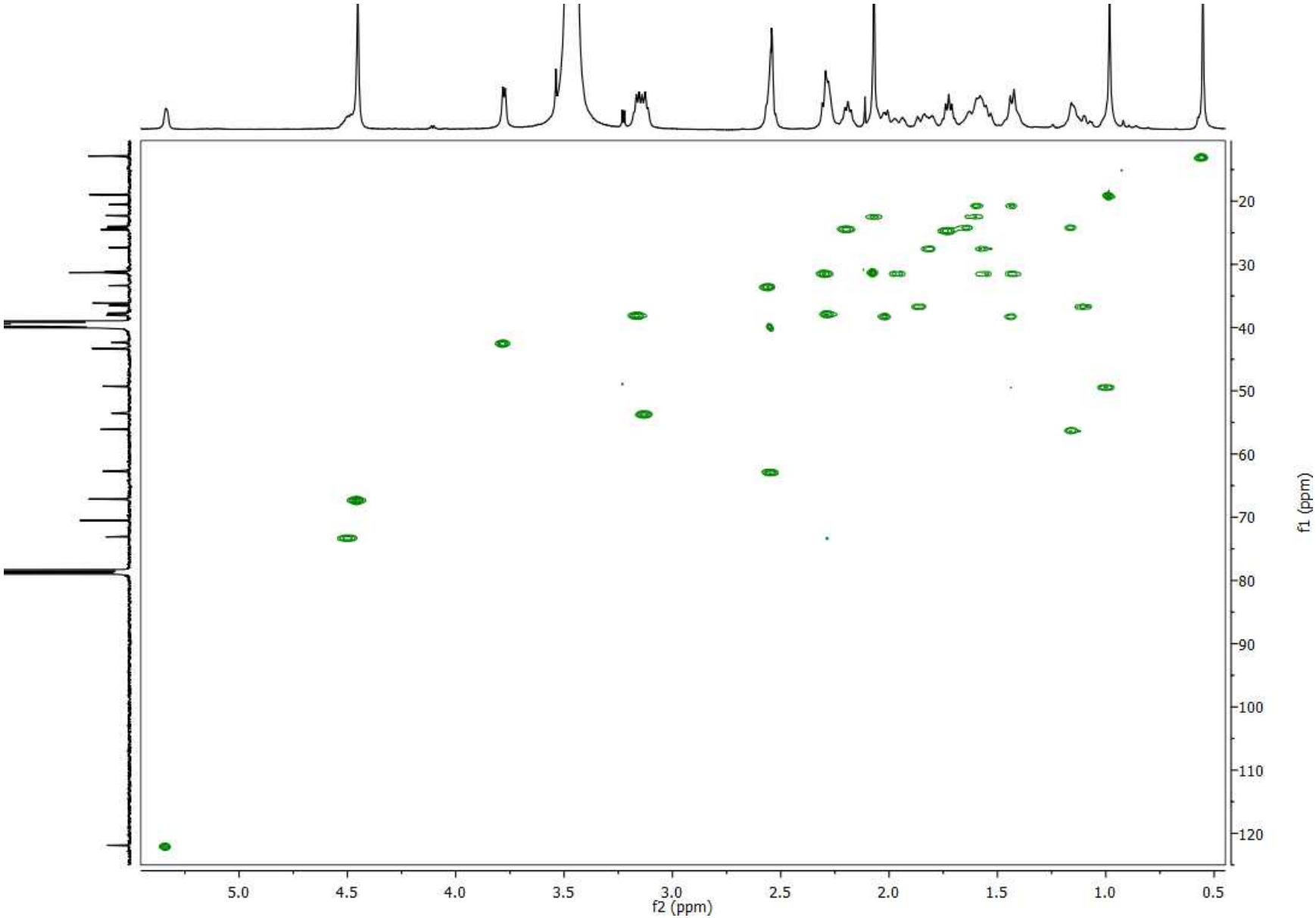
^{13}C NMR spectrum of thread **3a** in [D_6]DMSO



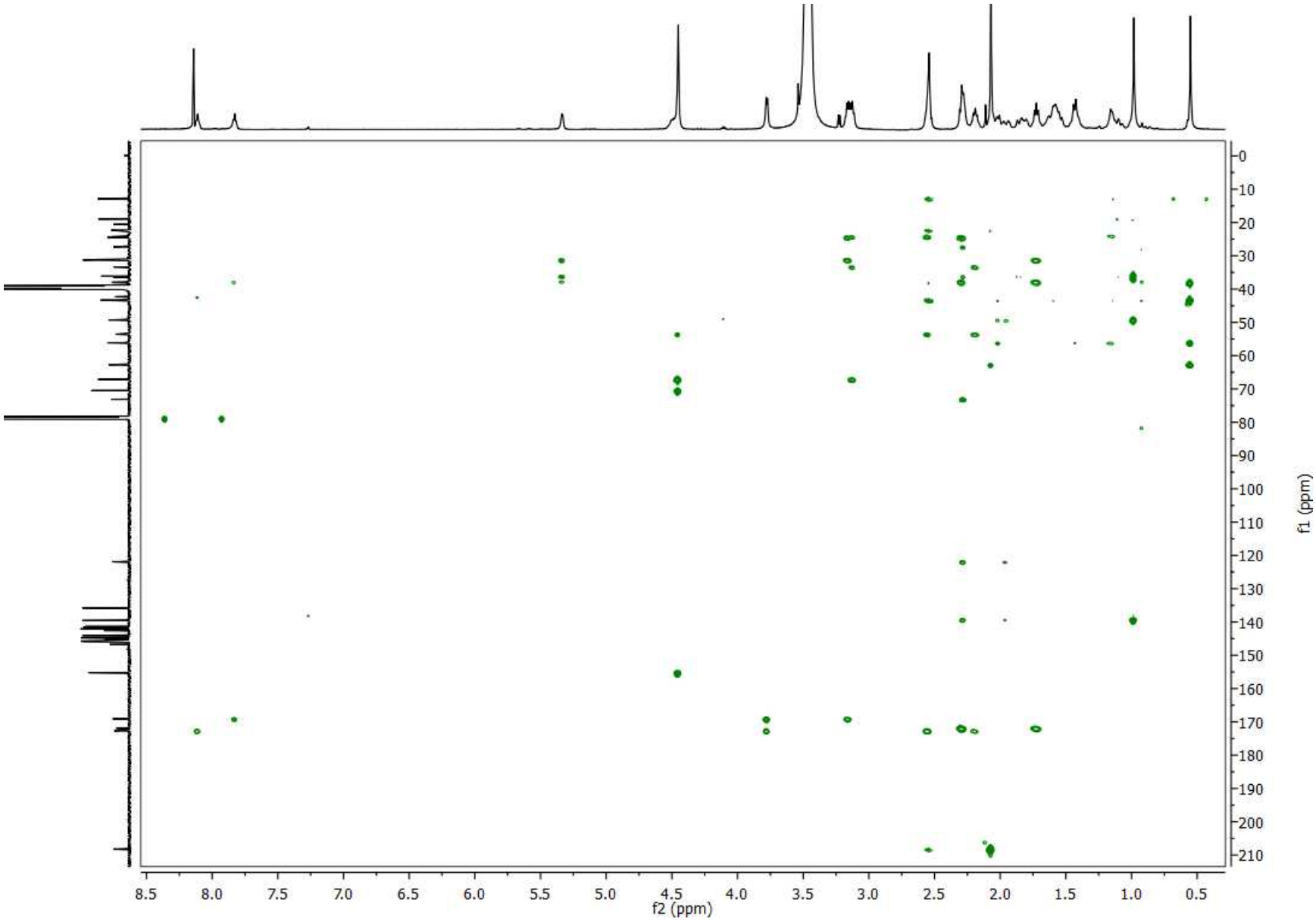
COSY spectrum of thread **3a** in [D_6]DMSO



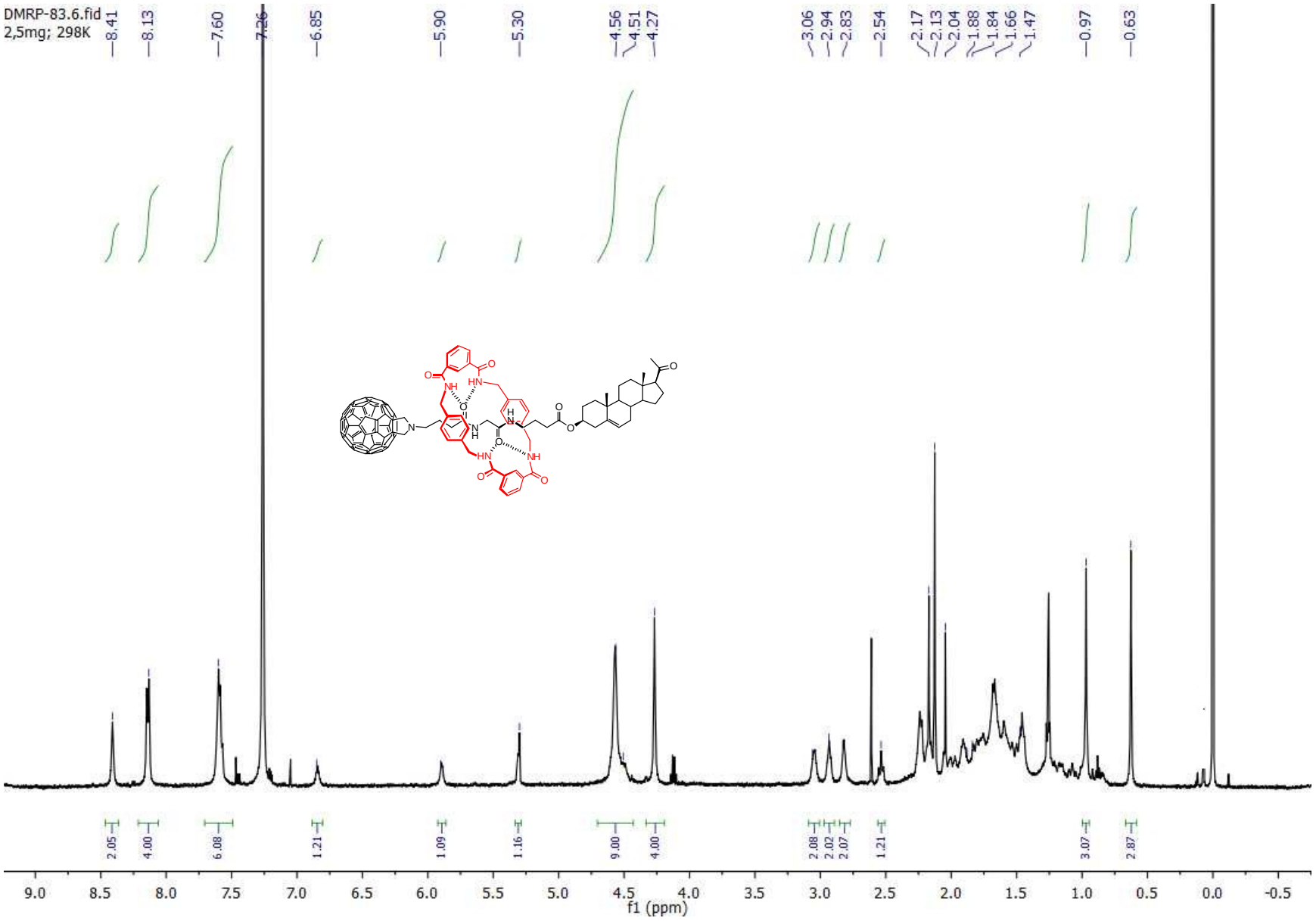
HSQC spectrum of thread **3a** in [D_6]DMSO



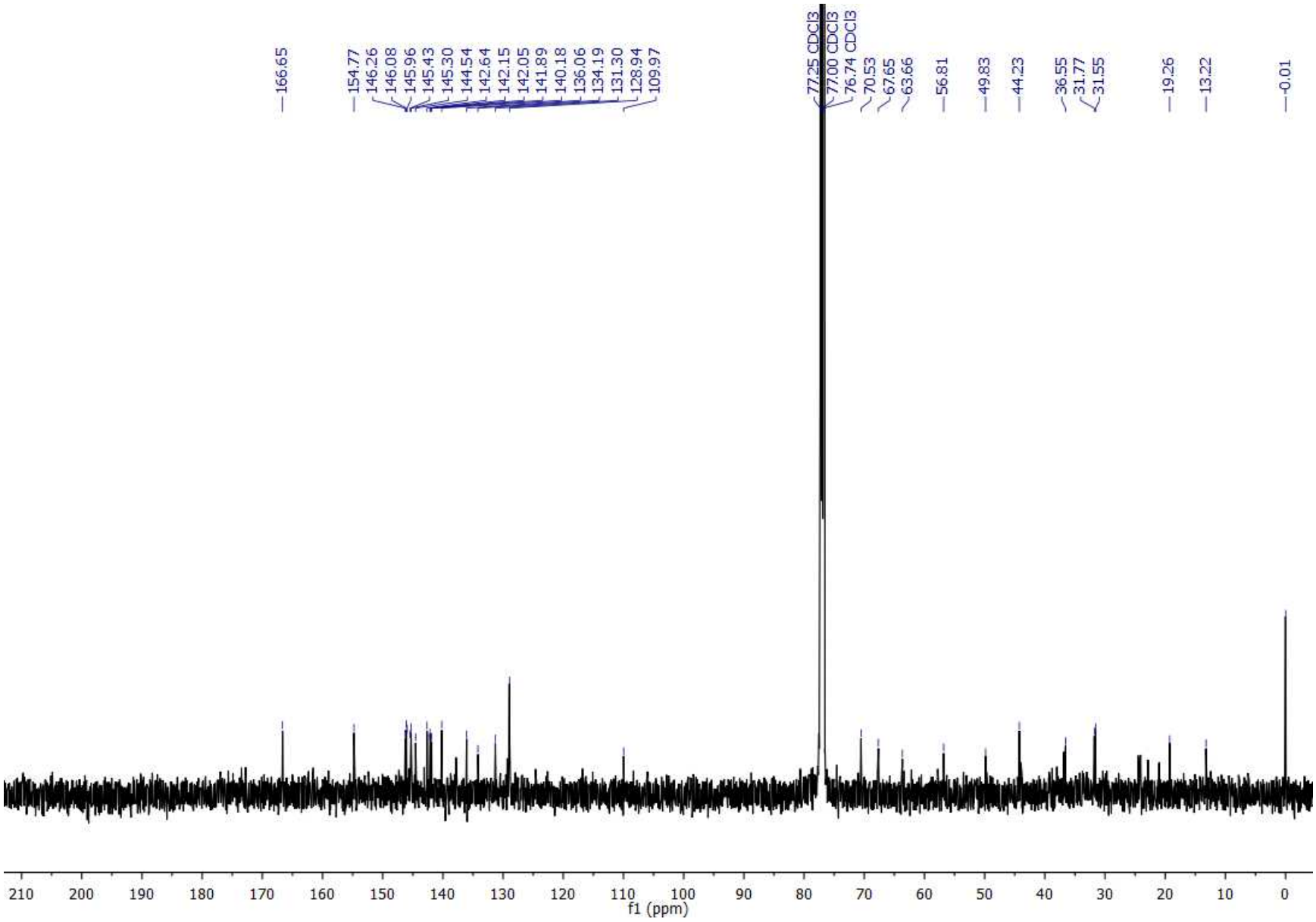
HMBC spectrum of thread **3a** in [D_6]DMSO



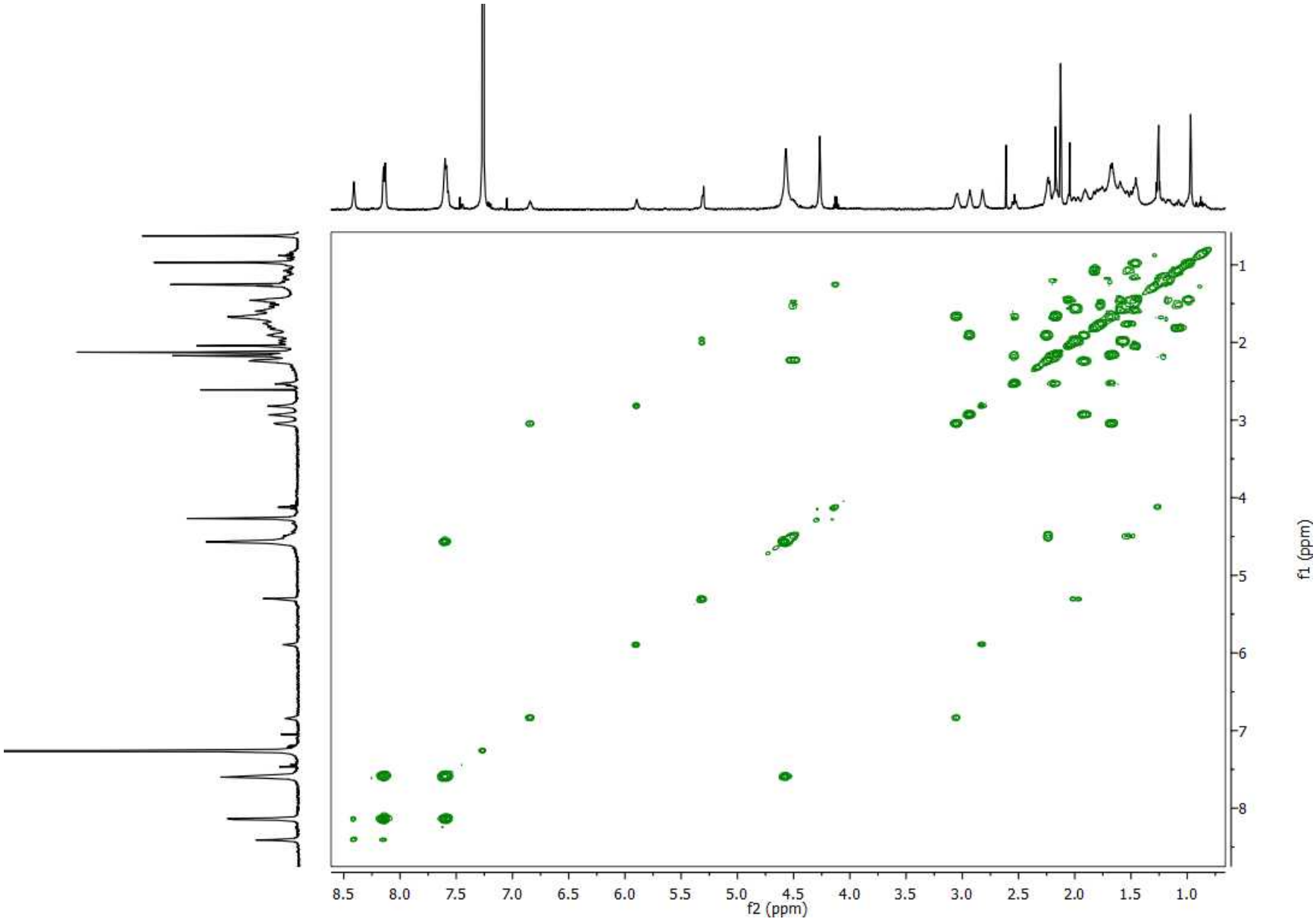
^{1}H NMR spectrum of rotaxane **4a** in $CDCl_3$



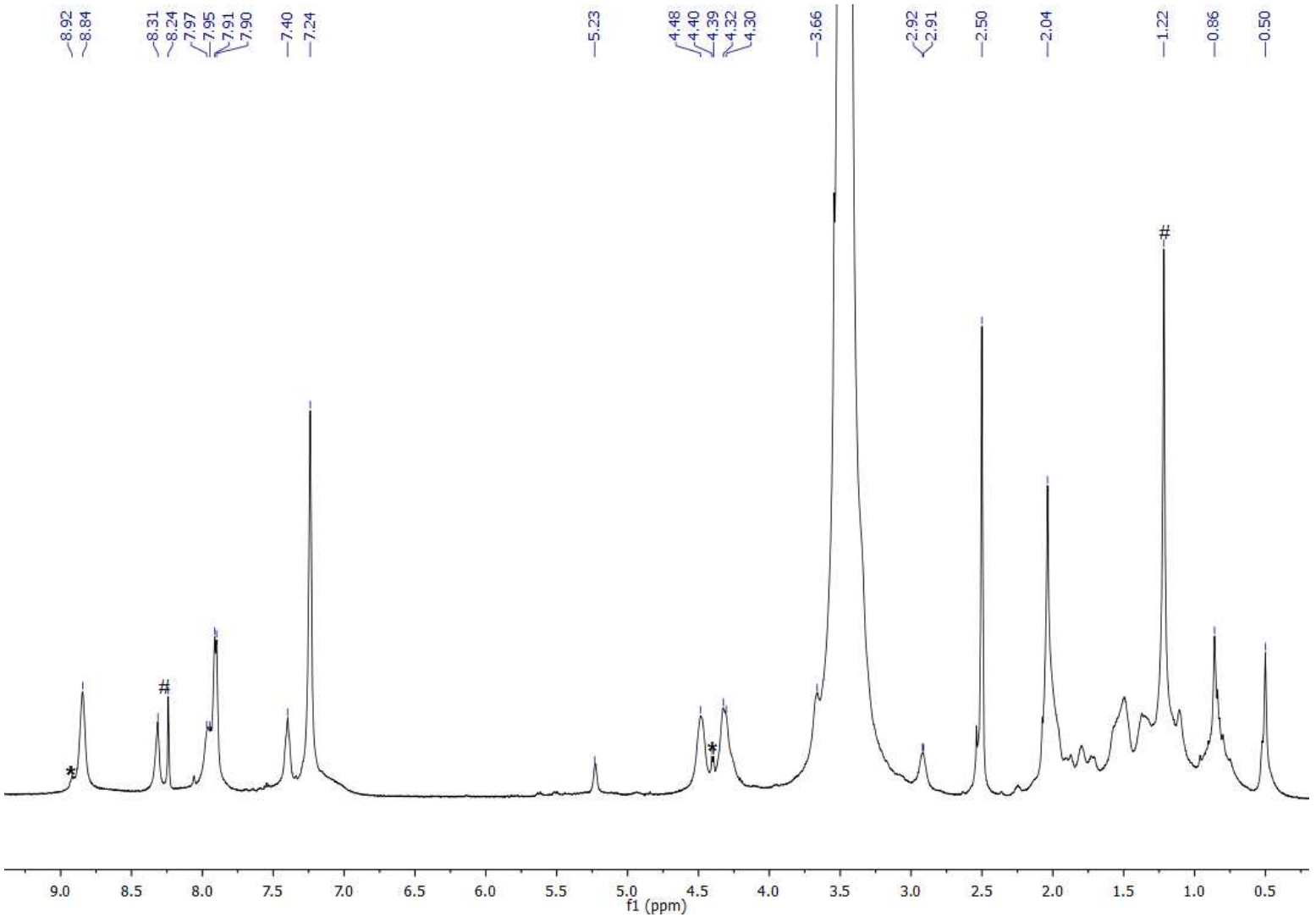
^{13}C NMR spectrum of rotaxane **4a** in $CDCl_3$



COSY spectrum of rotaxane **4a** in $CDCl_3$

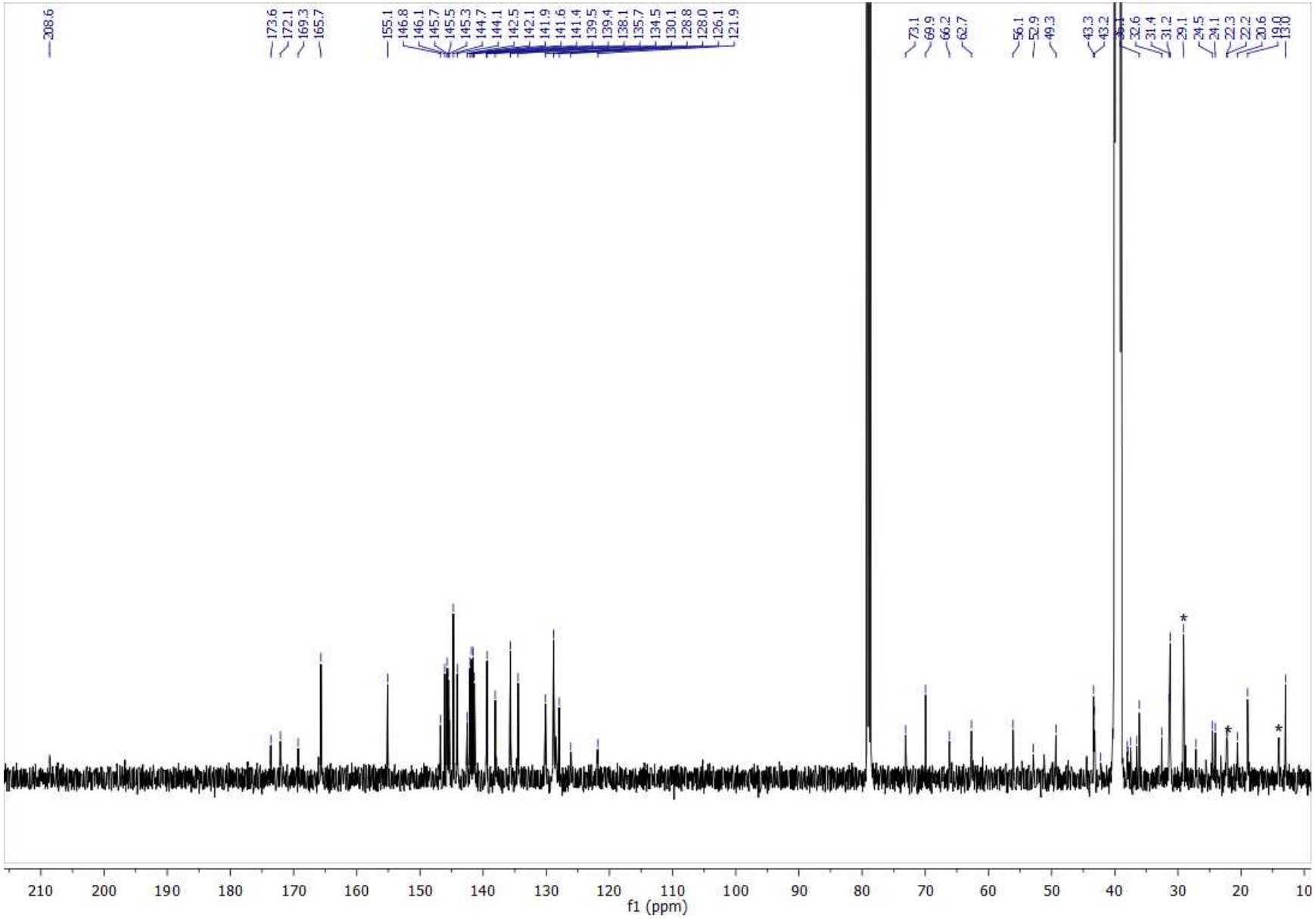


^{1}H NMR spectrum of rotaxane **4a** in [D_6]DMSO

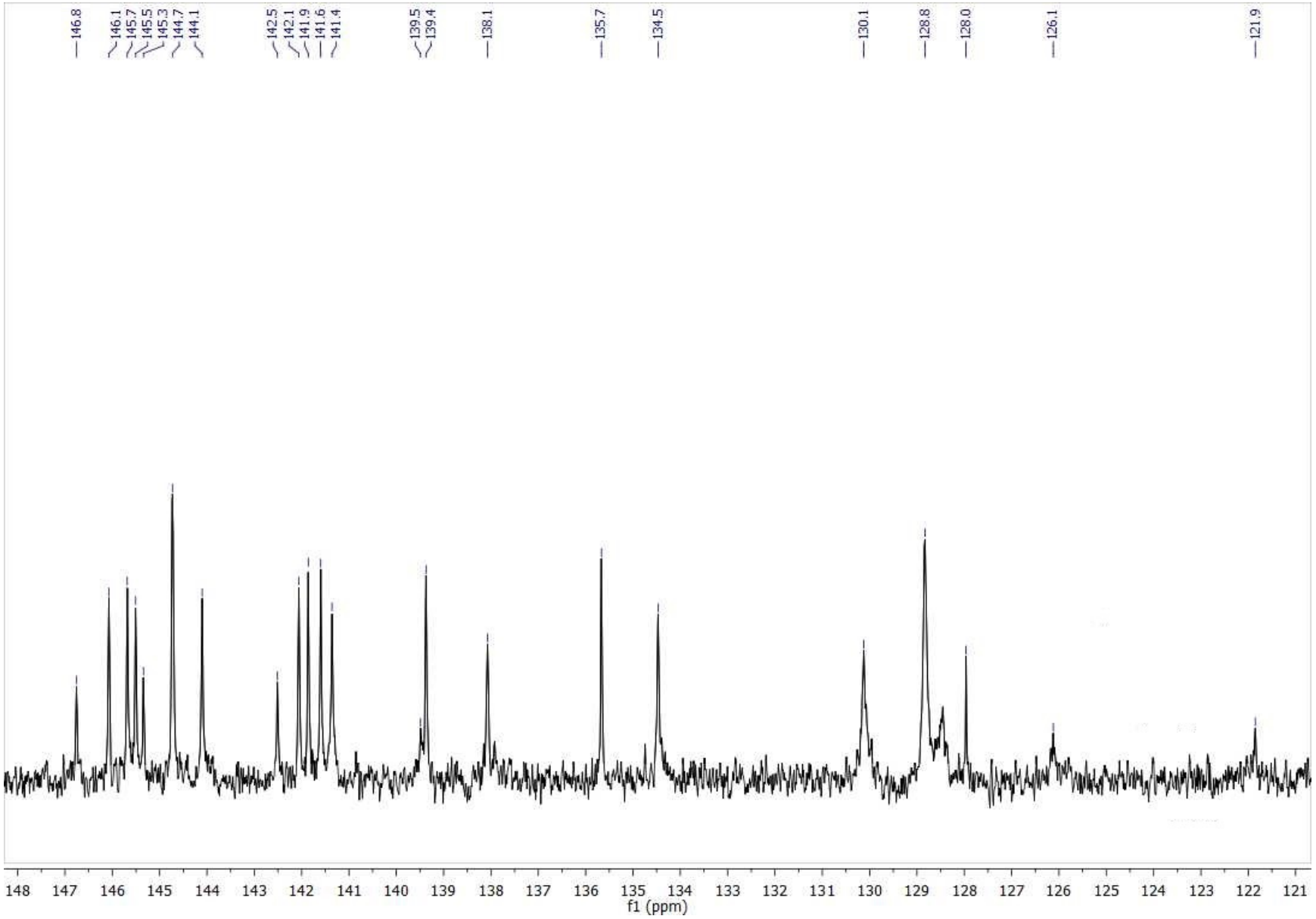


*Free macrocycle signals; # Solvent signals

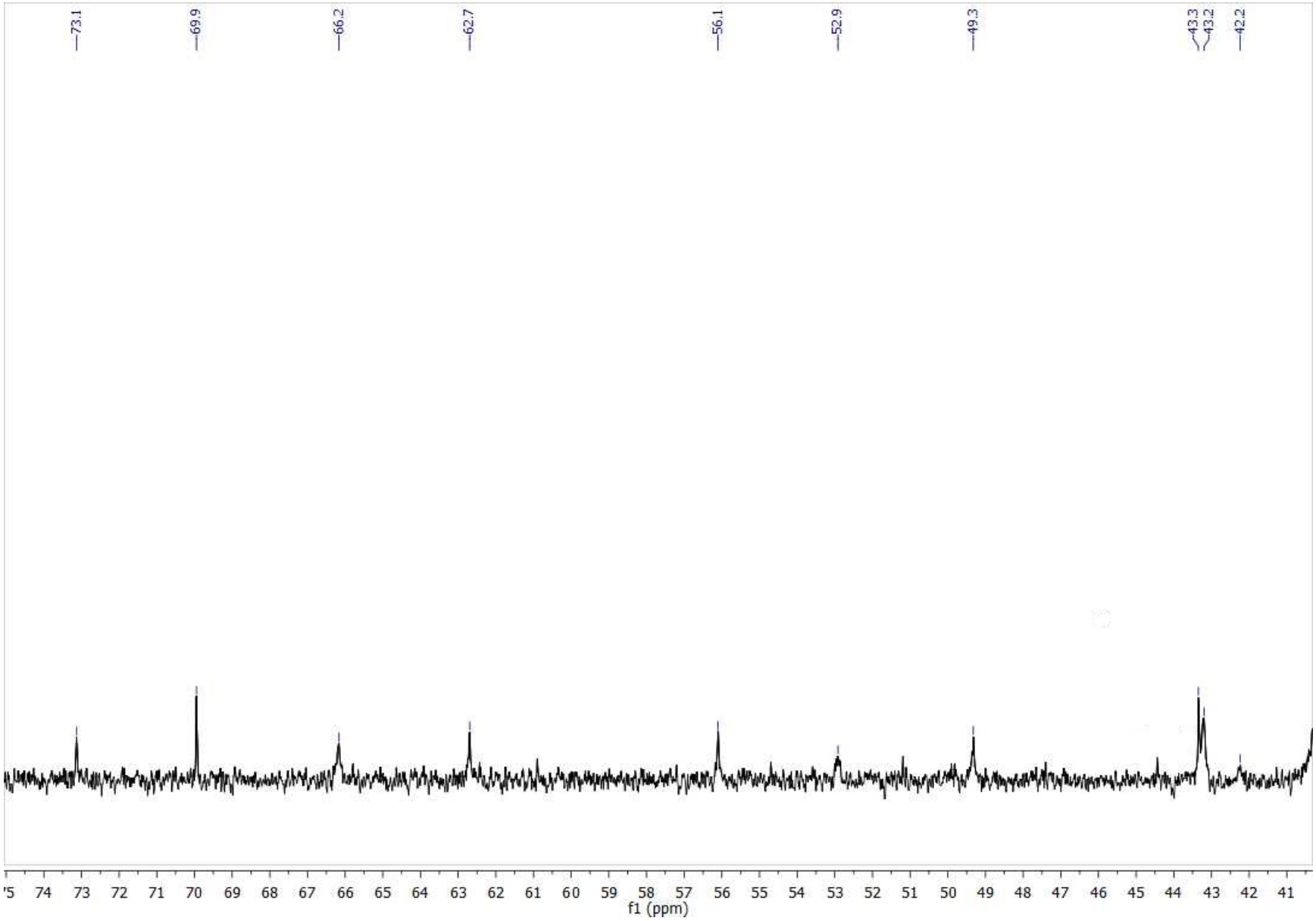
^{13}C NMR spectrum of rotaxane **4a** in [D_6]DMSO



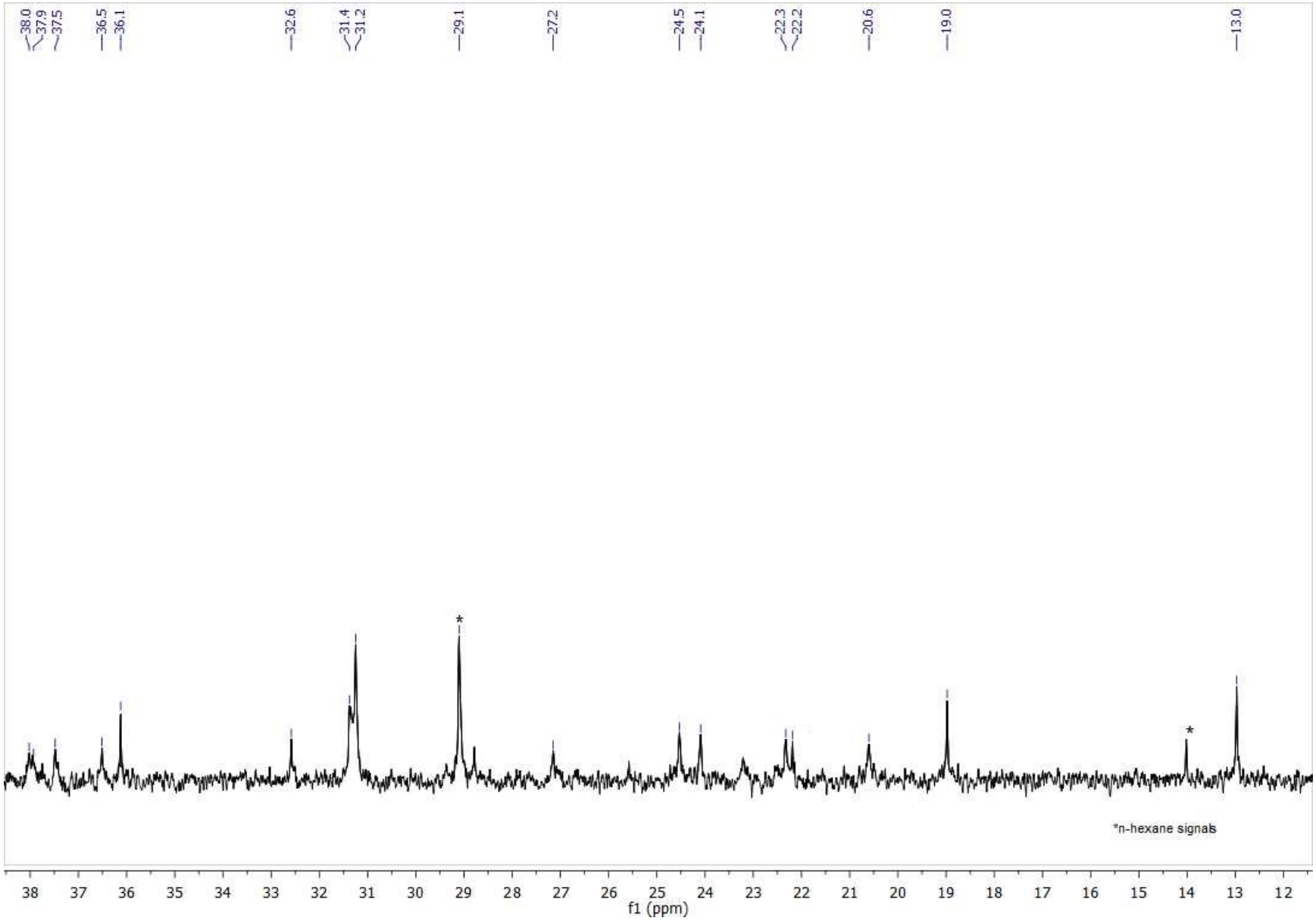
Expanded part of ^{13}C NMR spectrum of rotaxane **4a** in [D_6]DMSO



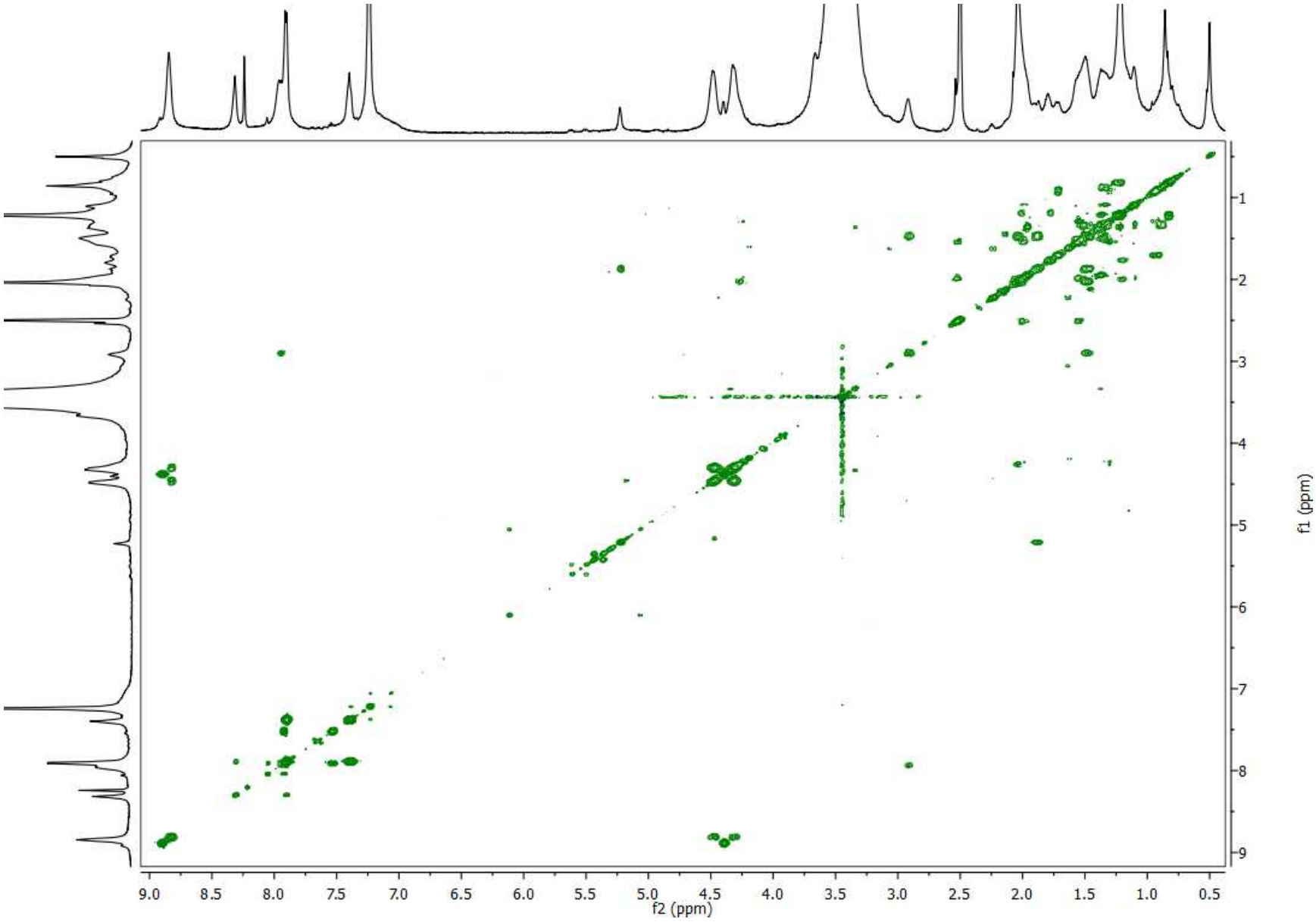
Expanded part of ^{13}C NMR spectrum of rotaxane **4a** in $[D_6]$DMSO



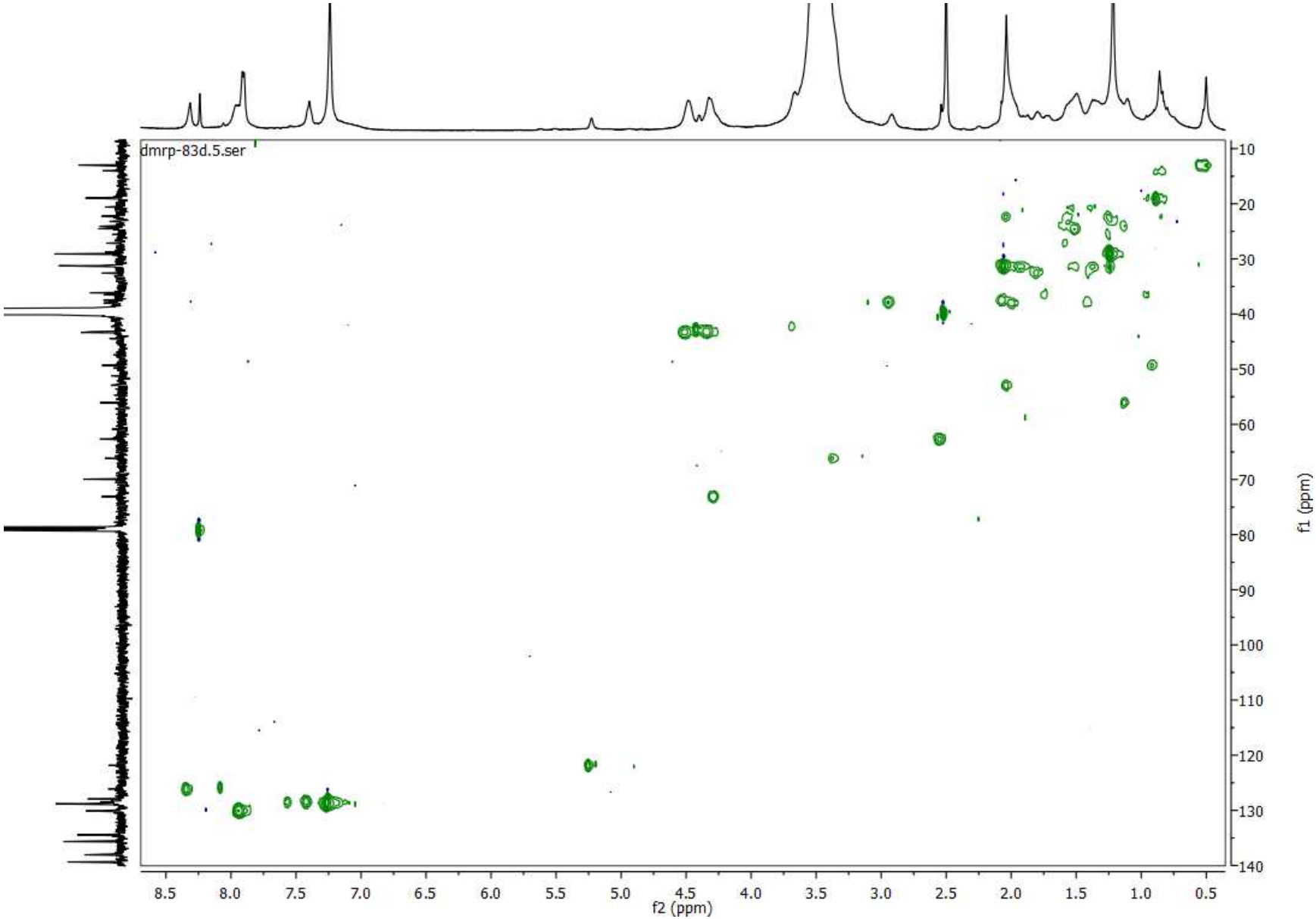
Expanded part of ^{13}C NMR spectrum of rotaxane **4a** in [D_6]DMSO



COSY spectrum of rotaxane **4a** in [D_6]DMSO



HSQC spectrum of rotaxane **4a** in [D_6]DMSO



Mass spectrum of rotaxane **4a**

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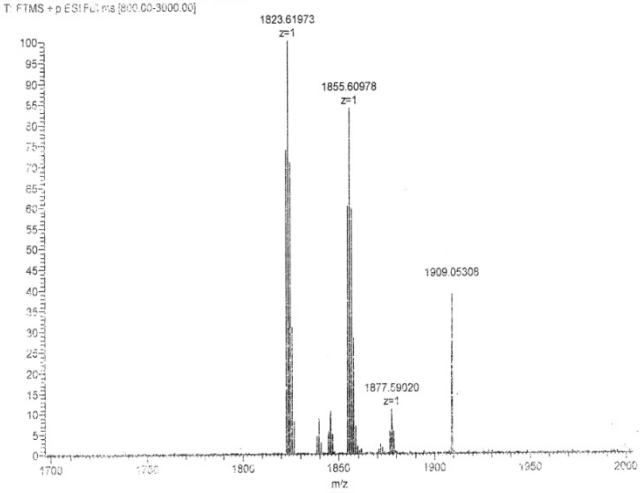
Maseni spektrometar: LTQ Orbitrap XL
Uzorak je uveden direktnom injekciom.
Tip jonizacije: Heated ESI (HESI)
Polaritet: Pozitivan
Analizator: Orbitrap

Uslovi snimanja :

Spray Voltage (kV):	4.2
Vaporizer Temp (°C):	50
Sheath Gas Flow Rate:	12.00
Aux Gas Flow Rate:	3.00
Sweep Gas Flow Rate:	0
Capillary Voltage (V):	42
Capillary Temp (°C):	275
Tube Lens (V):	140

DMRP-83
Raw file: O83041
Uzorak: DMRP-83 1ug/mL in MeOH (HPLC grade)



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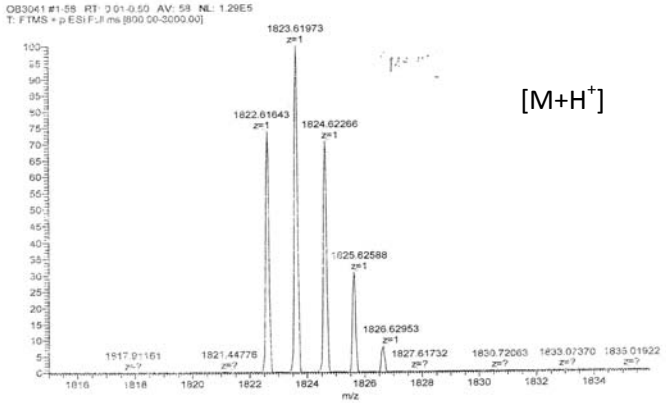
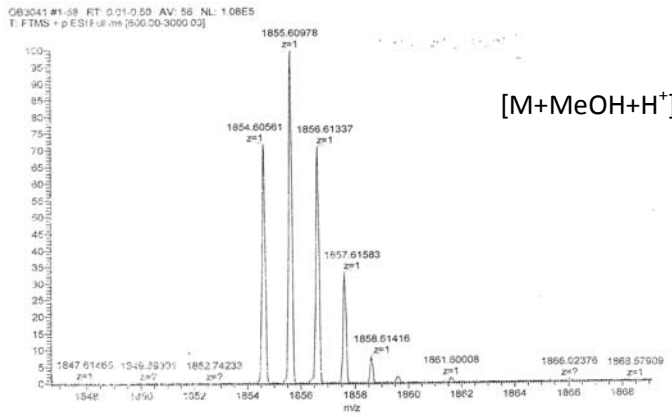


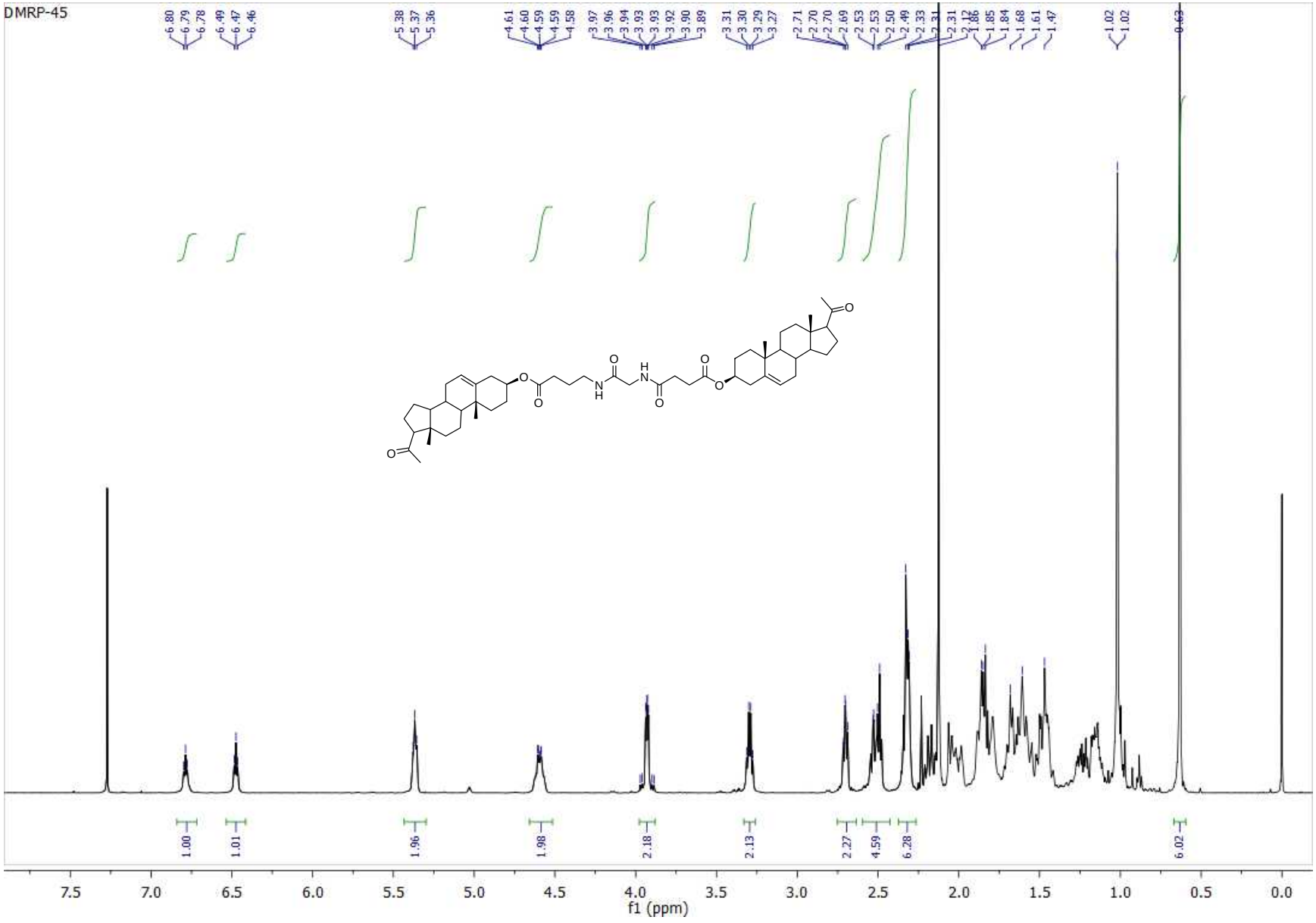
[M+H$^+$]

Zum uprosecenog spektra u m/z 1845 - 1860

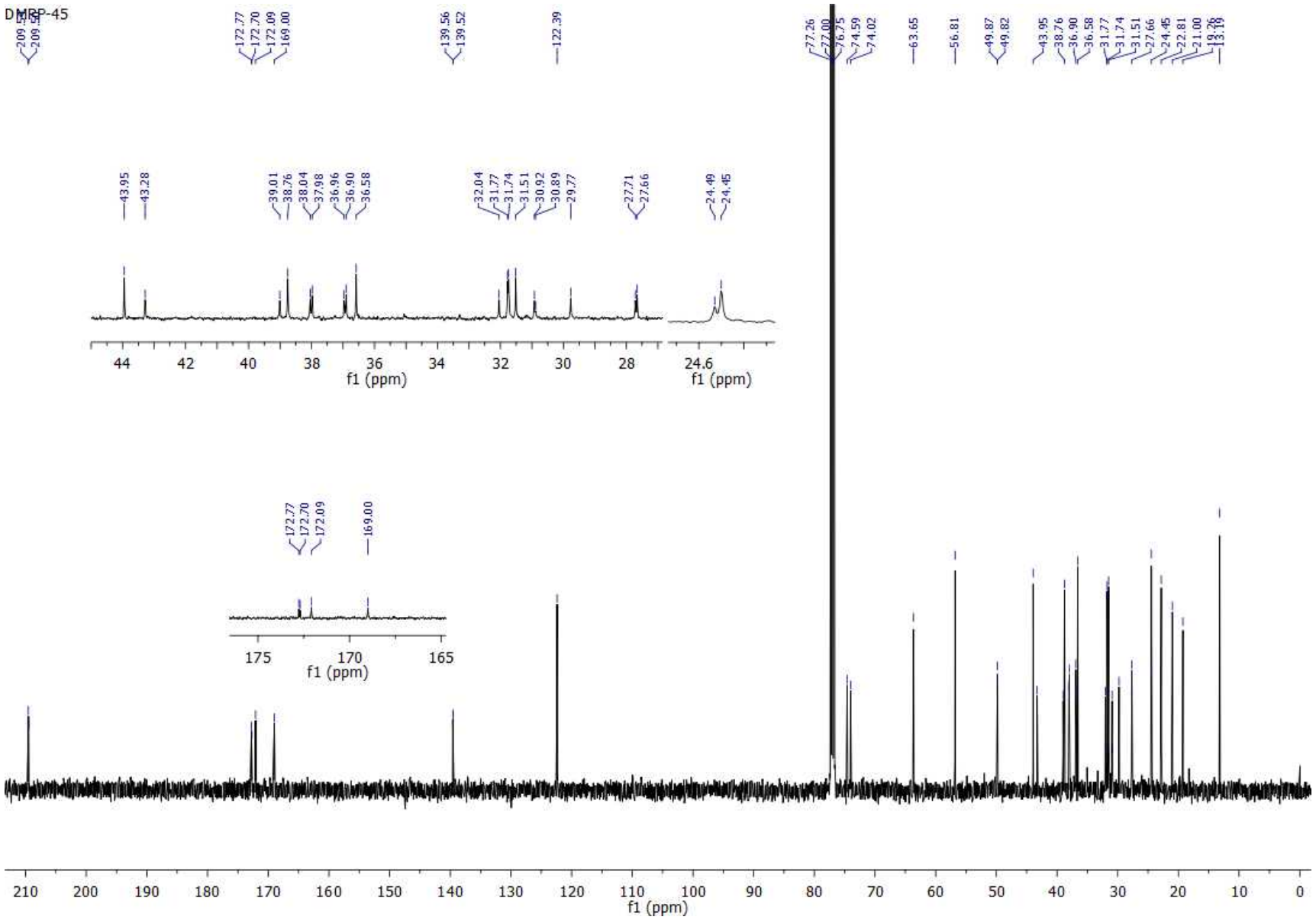


[M+MeOH+H$^+$]

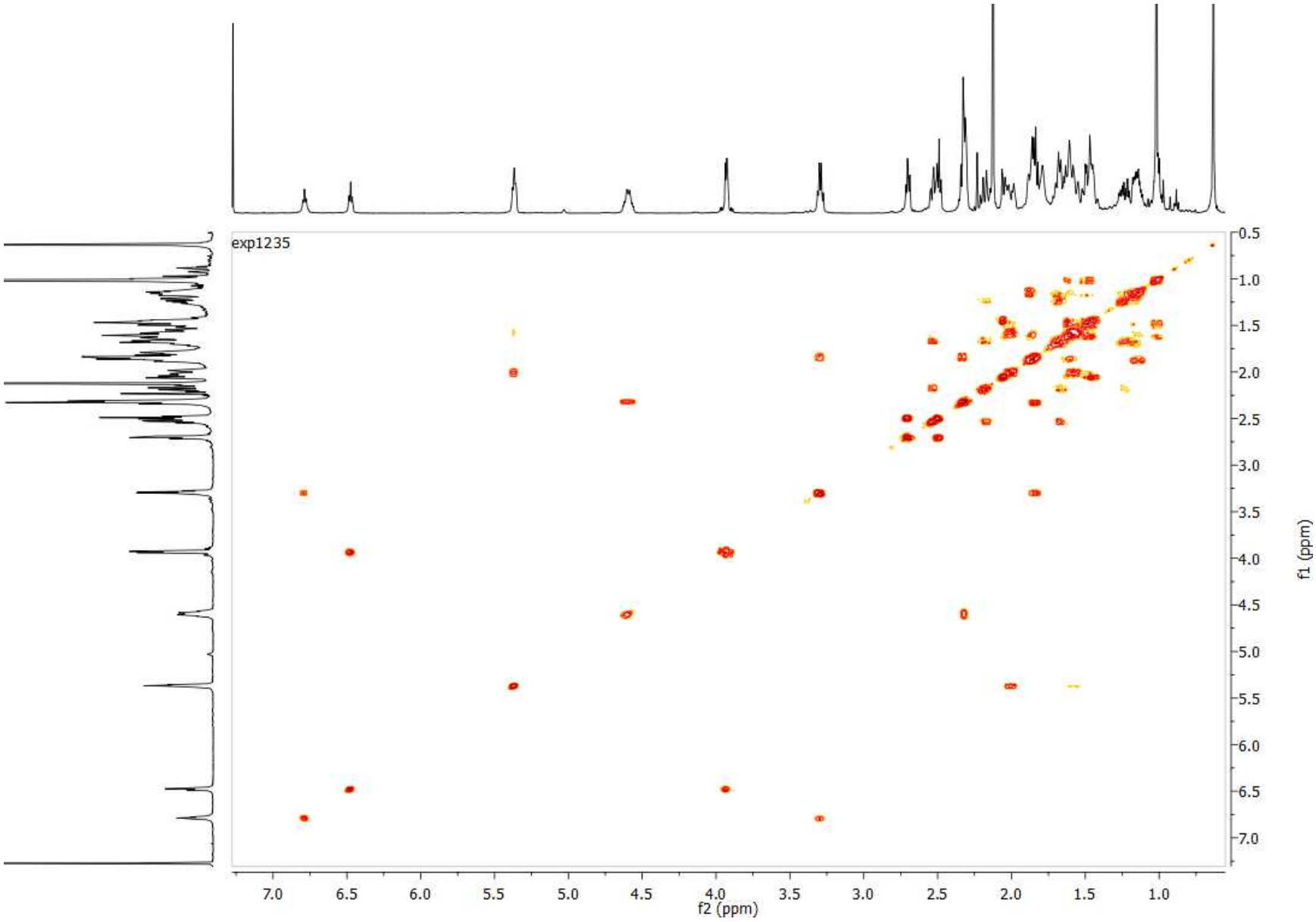
^{1}H NMR spectrum of thread **3b** in $CDCl_3$



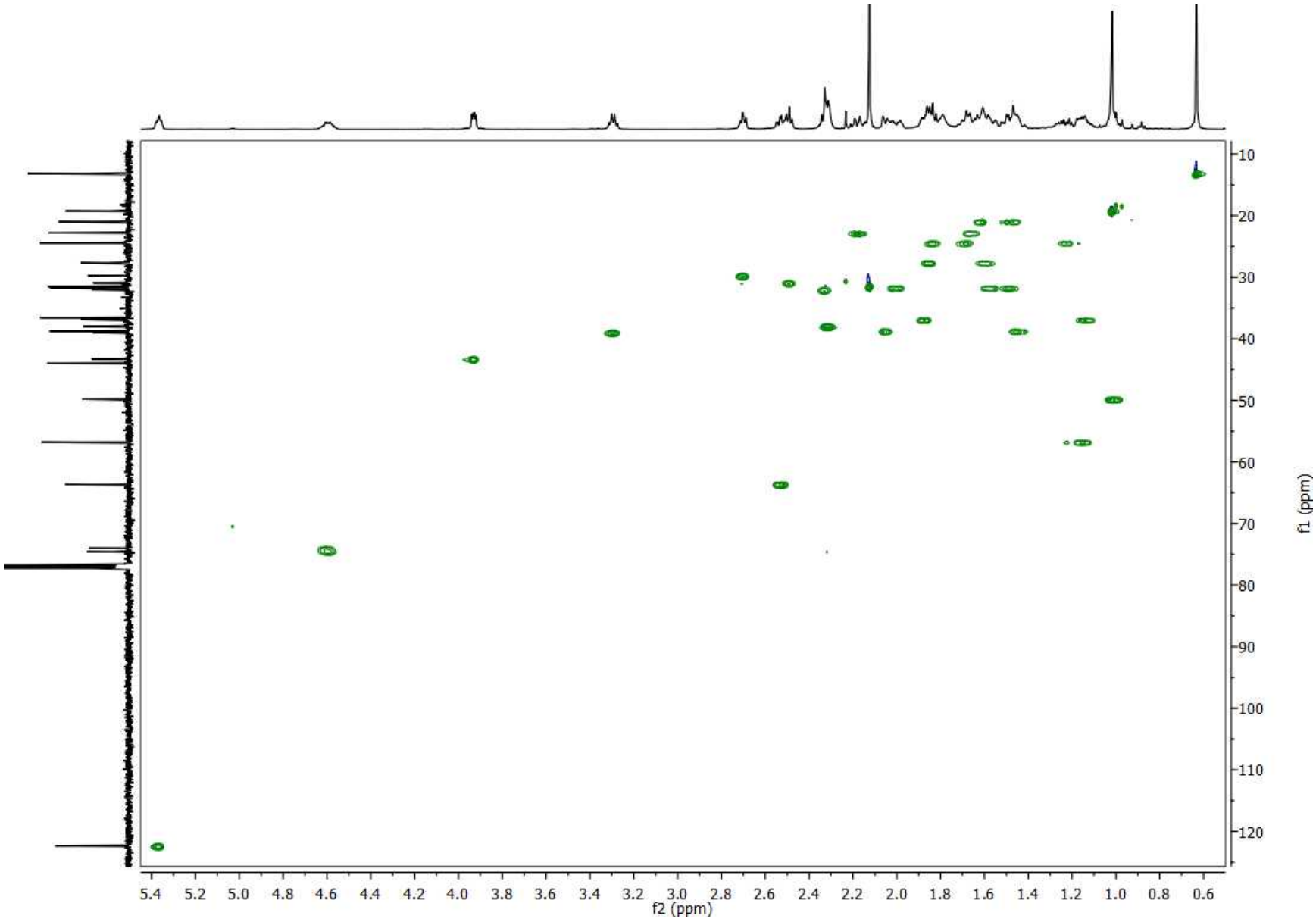
^{13}C NMR spectrum of thread **3b** in $CDCl_3$



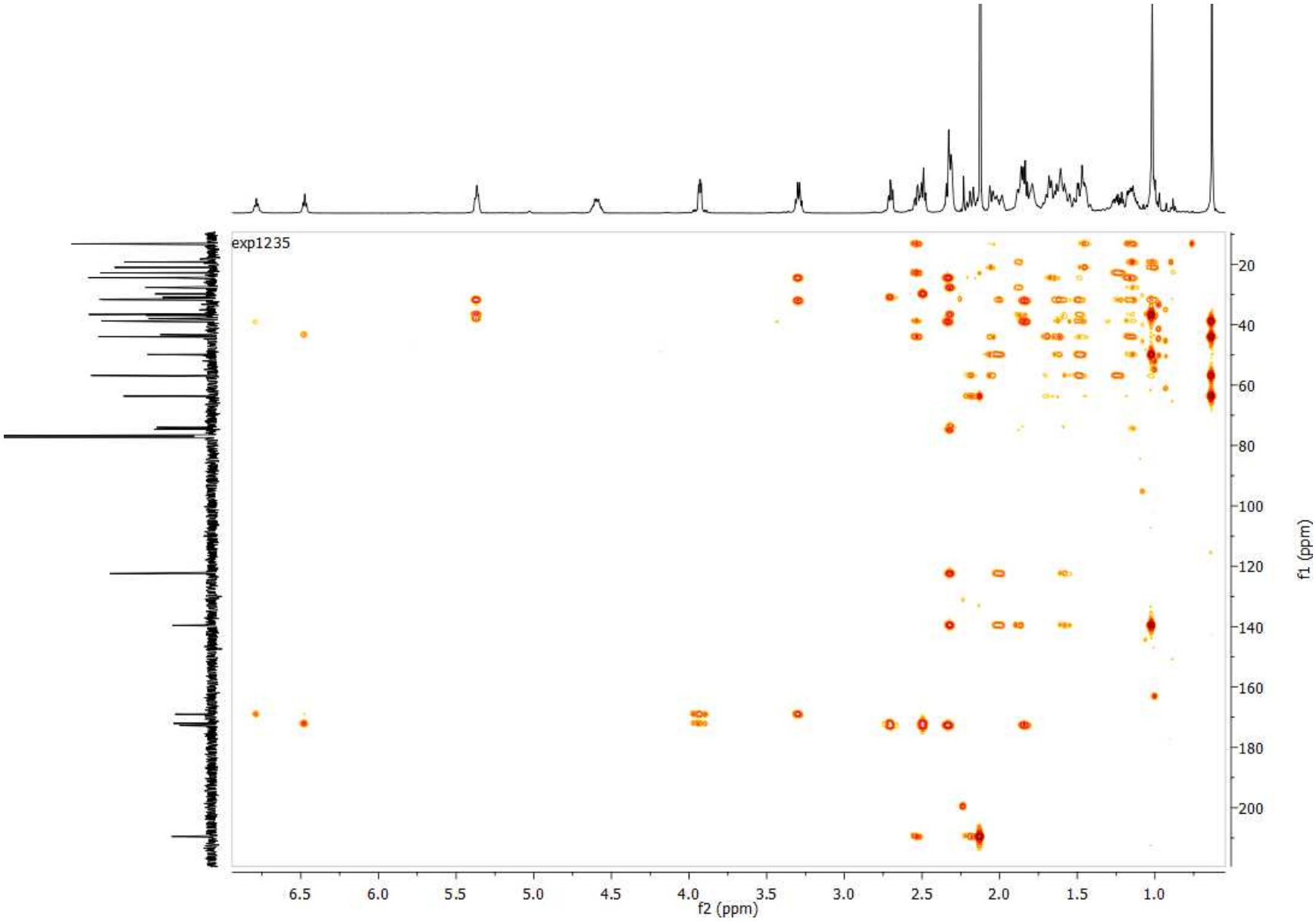
COSY spectrum of thread **3b** in $CDCl_3$



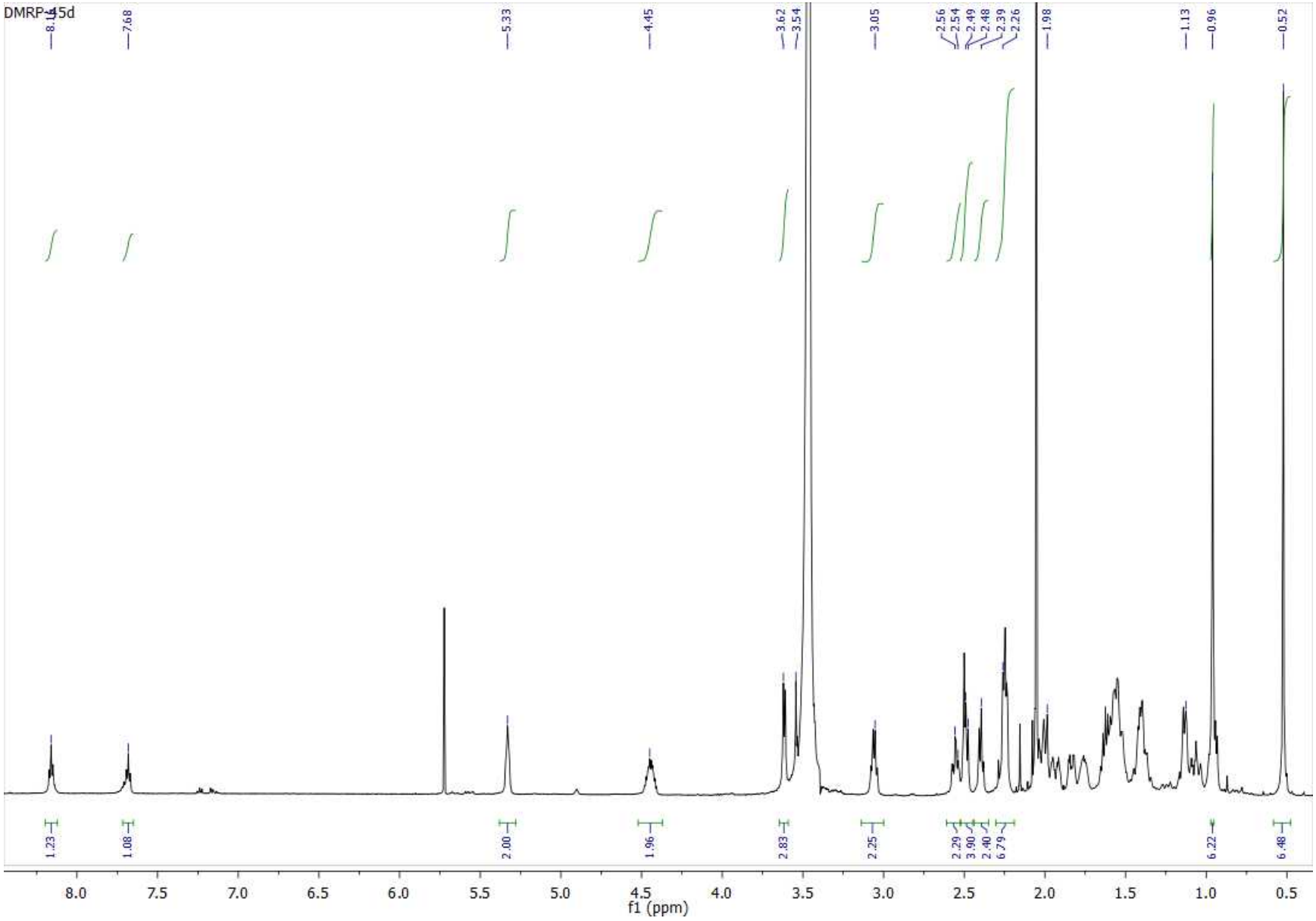
HSQC spectrum of thread **3b** in $CDCl_3$



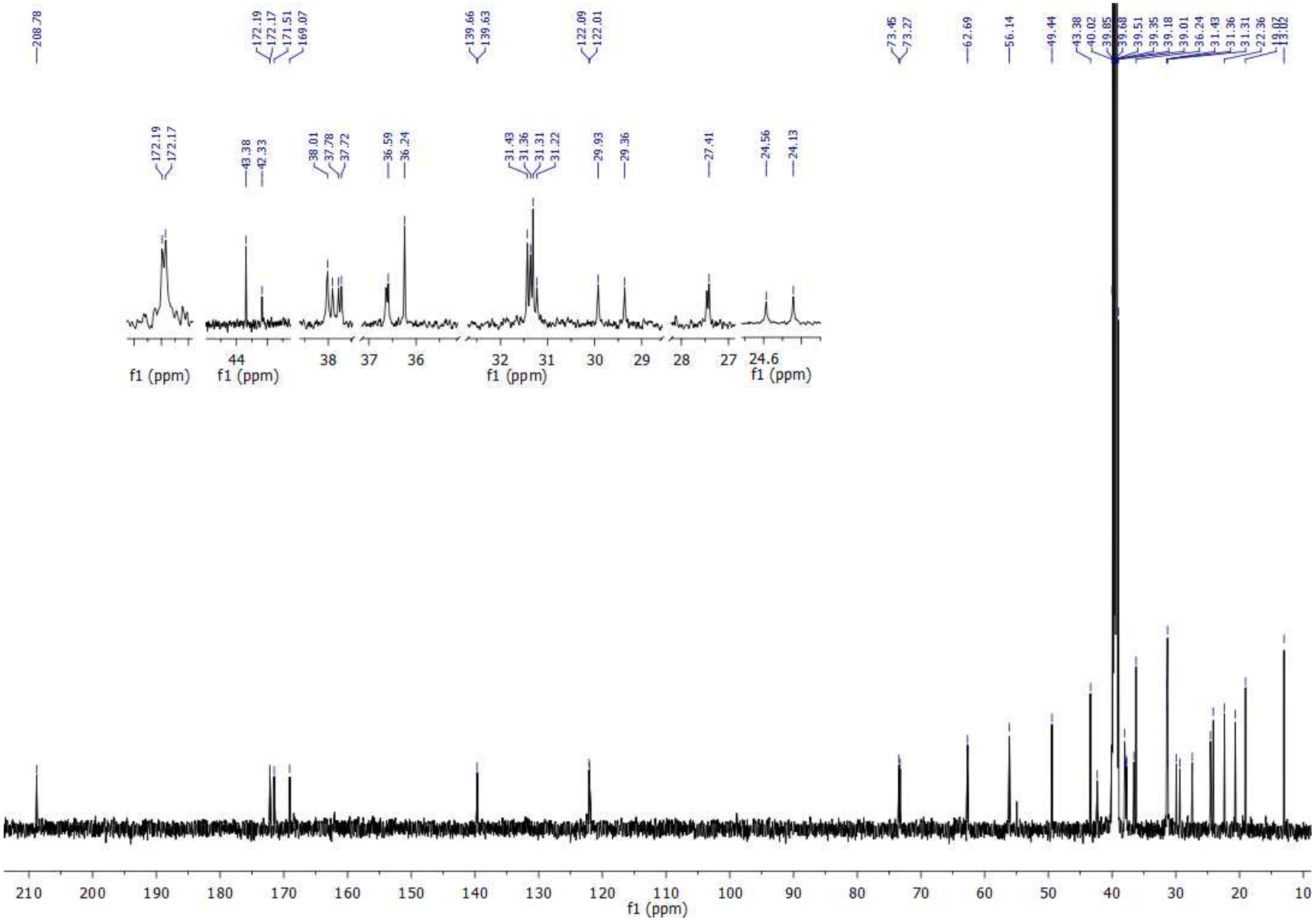
HMBC spectrum of thread **3b** in $CDCl_3$



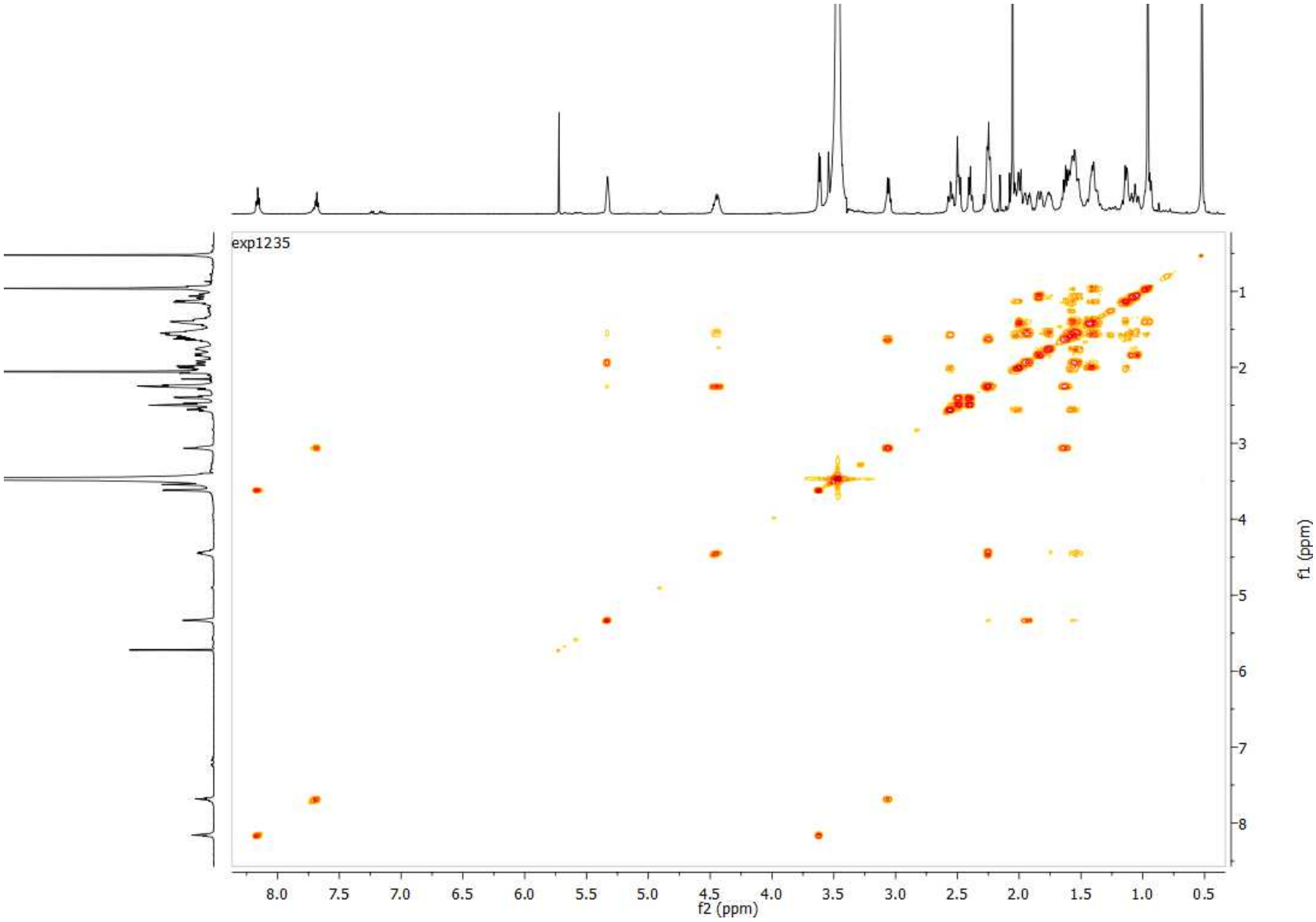
^{1}H NMR spectrum of thread **3b** in [D_6]DMSO



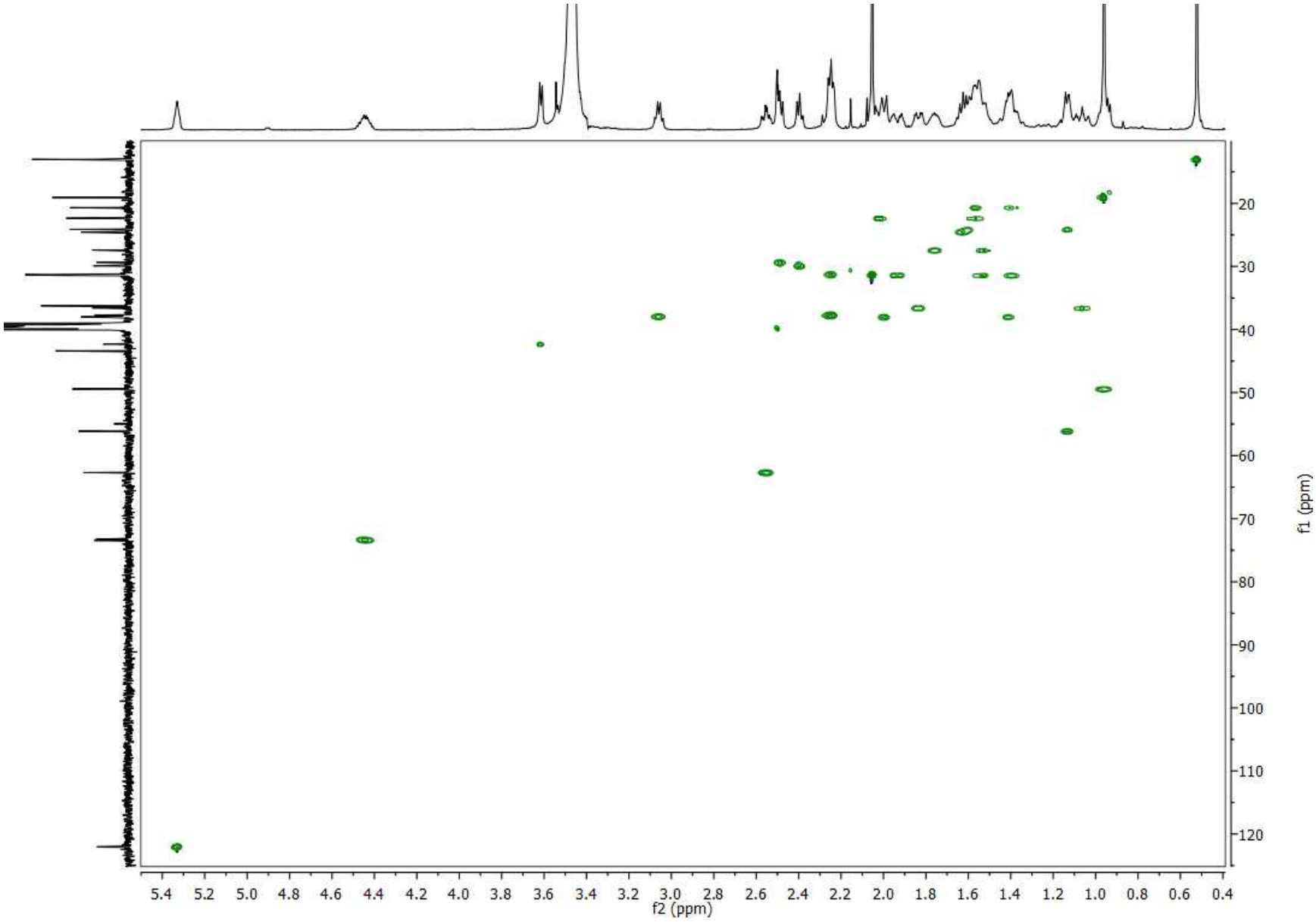
^{13}C NMR spectrum of thread **3b** in [D_6]DMSO



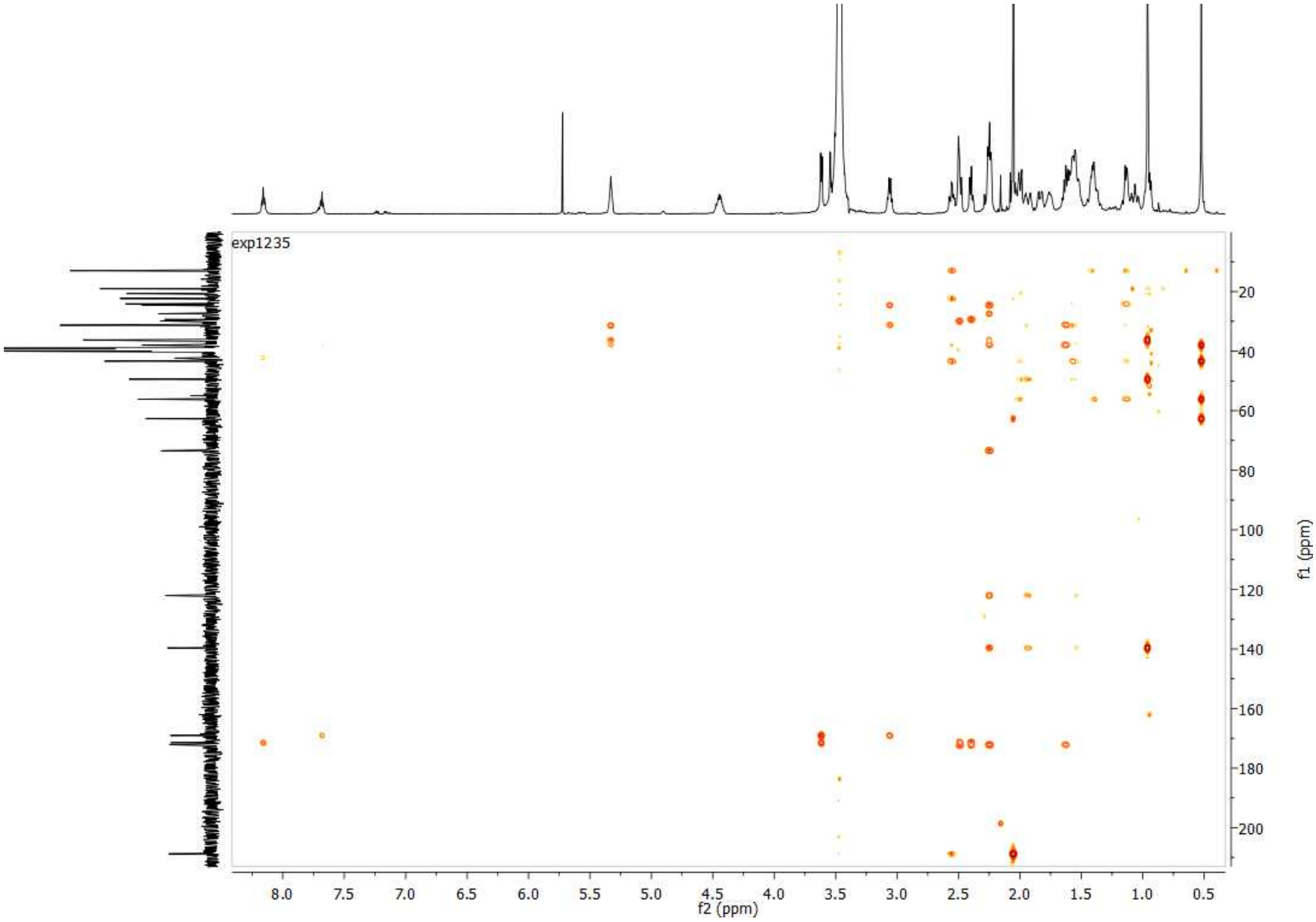
COSY spectrum of thread **3b** in [D_6]DMSO



HSQC spectrum of thread **3b** in [D_6]DMSO



HMBC spectrum of thread **3b** in [D_6]DMSO



DMRP-45

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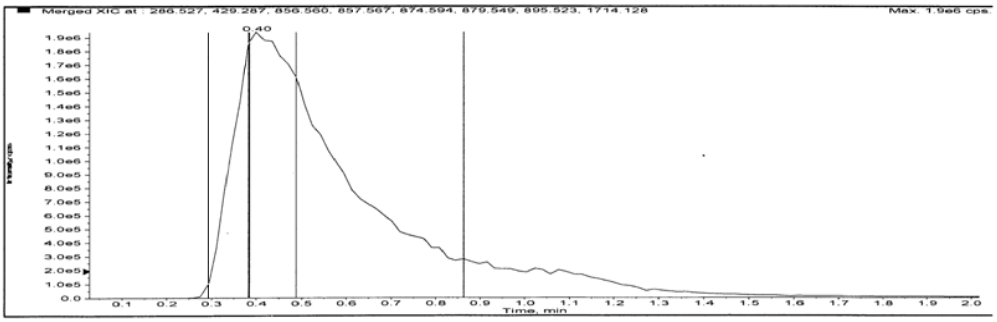
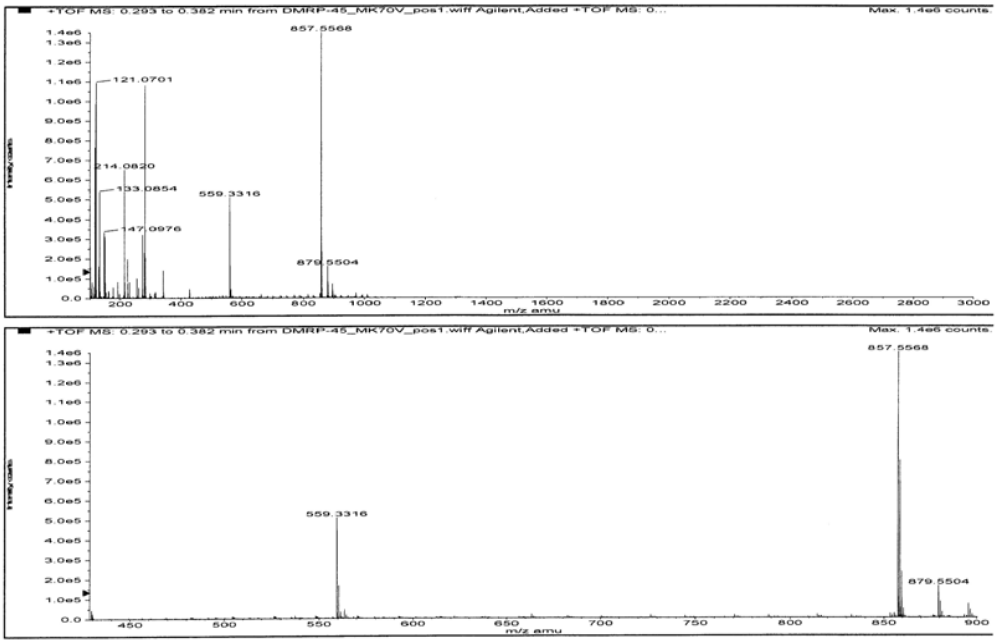
Method: **d:\TOF_Data\damethods\Night_Seq_Comp_ident1.anm\efc.xml**



Merged XIC, Period# : 1 Experiment# : 1

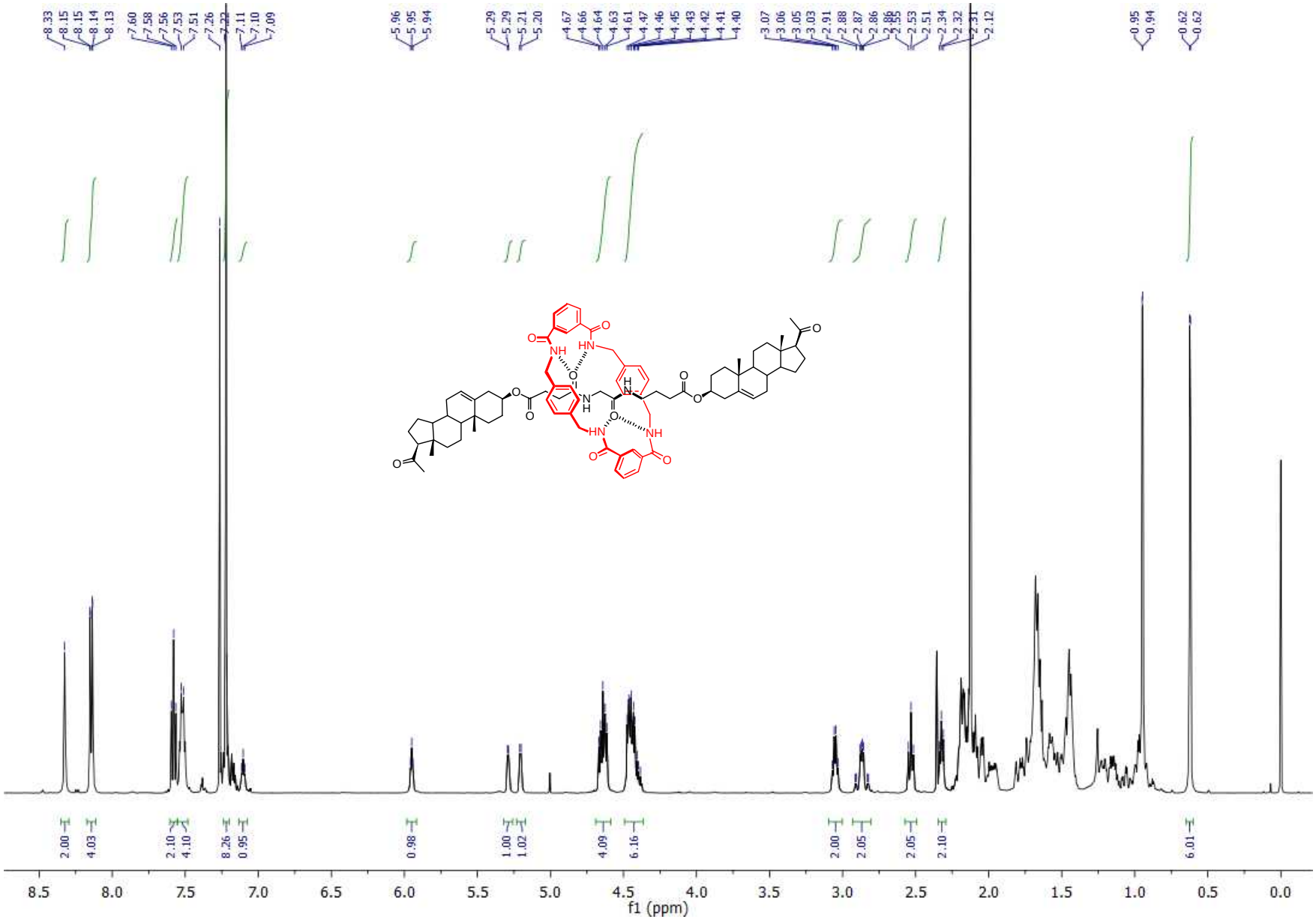


Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C52H76N2O8	--	856.56017	0.40	3.03214 E7	--

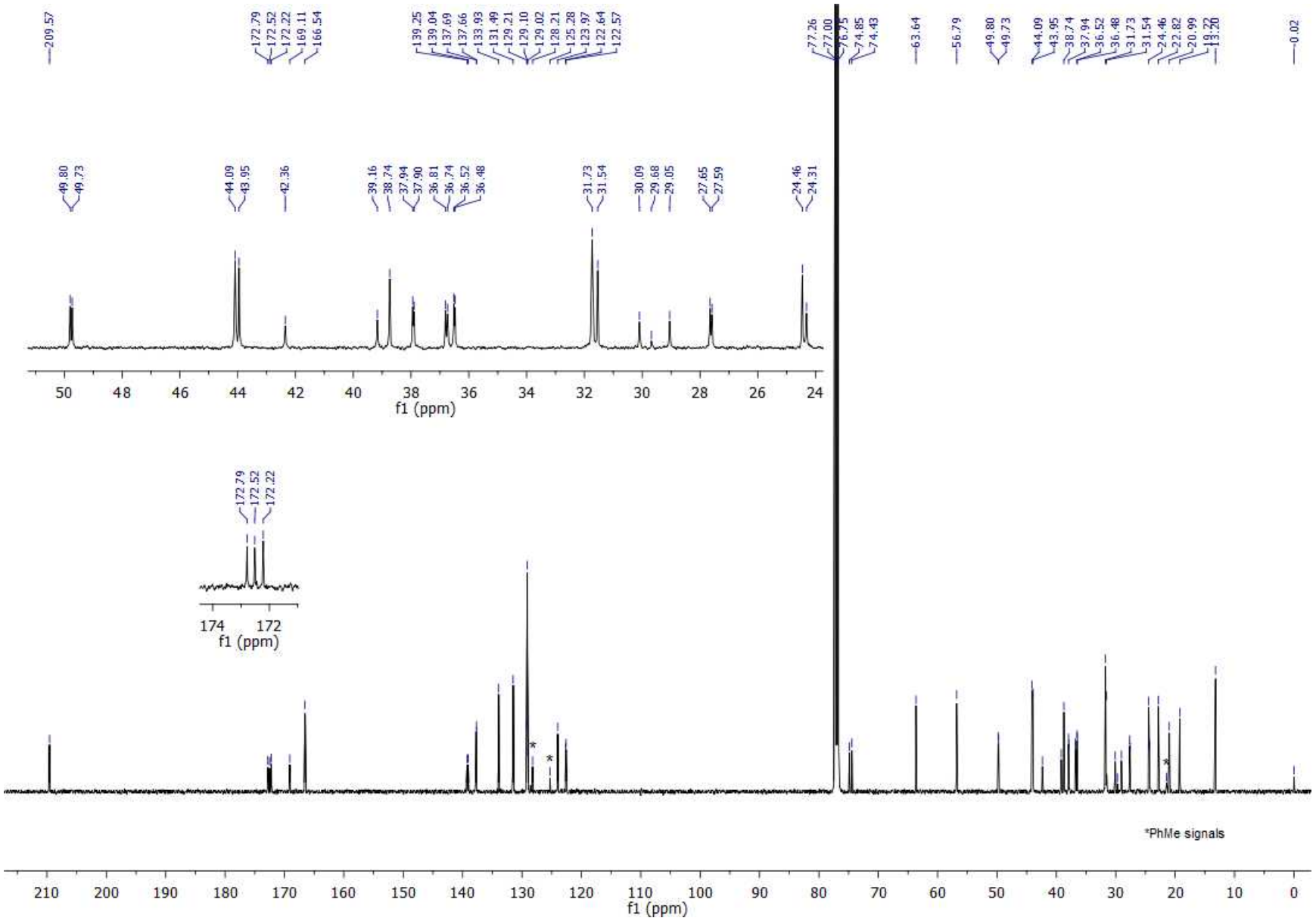
Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
[M+2H]2+	47307.66	429.28736	429.28751	0.15381	0.36	--
[M+H]+	1379227.57	857.56744	857.56645	-0.99521	-1.16	--
[M+Na]+	161903.47	879.54939	879.54987	0.47865	0.54	--
[M+K]+	72865.83	895.52333	895.52382	0.49343	0.55	--

Mass spectrum of thread **3b**

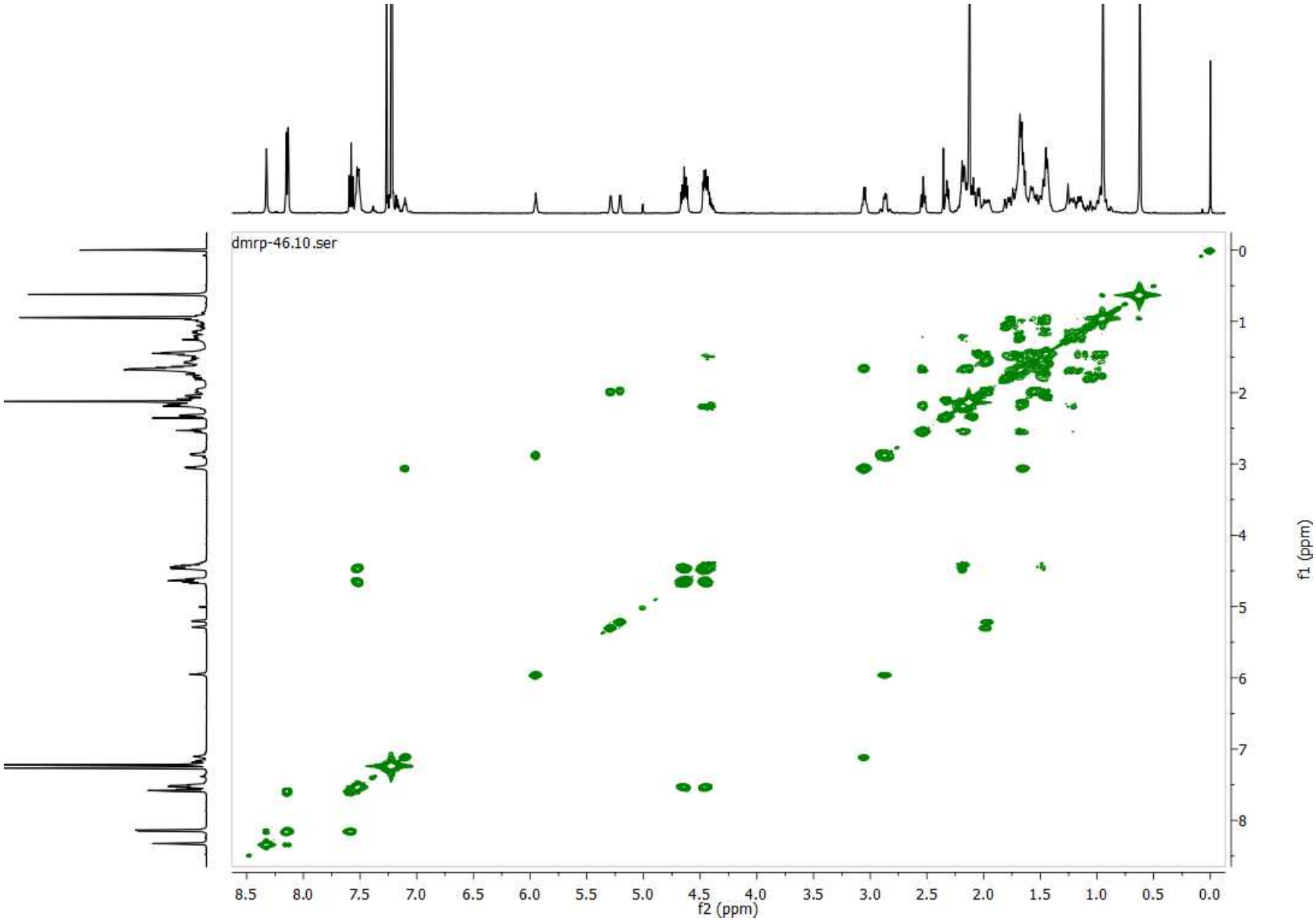
^{1}H NMR spectrum of rotaxane **4b** in $CDCl_3$



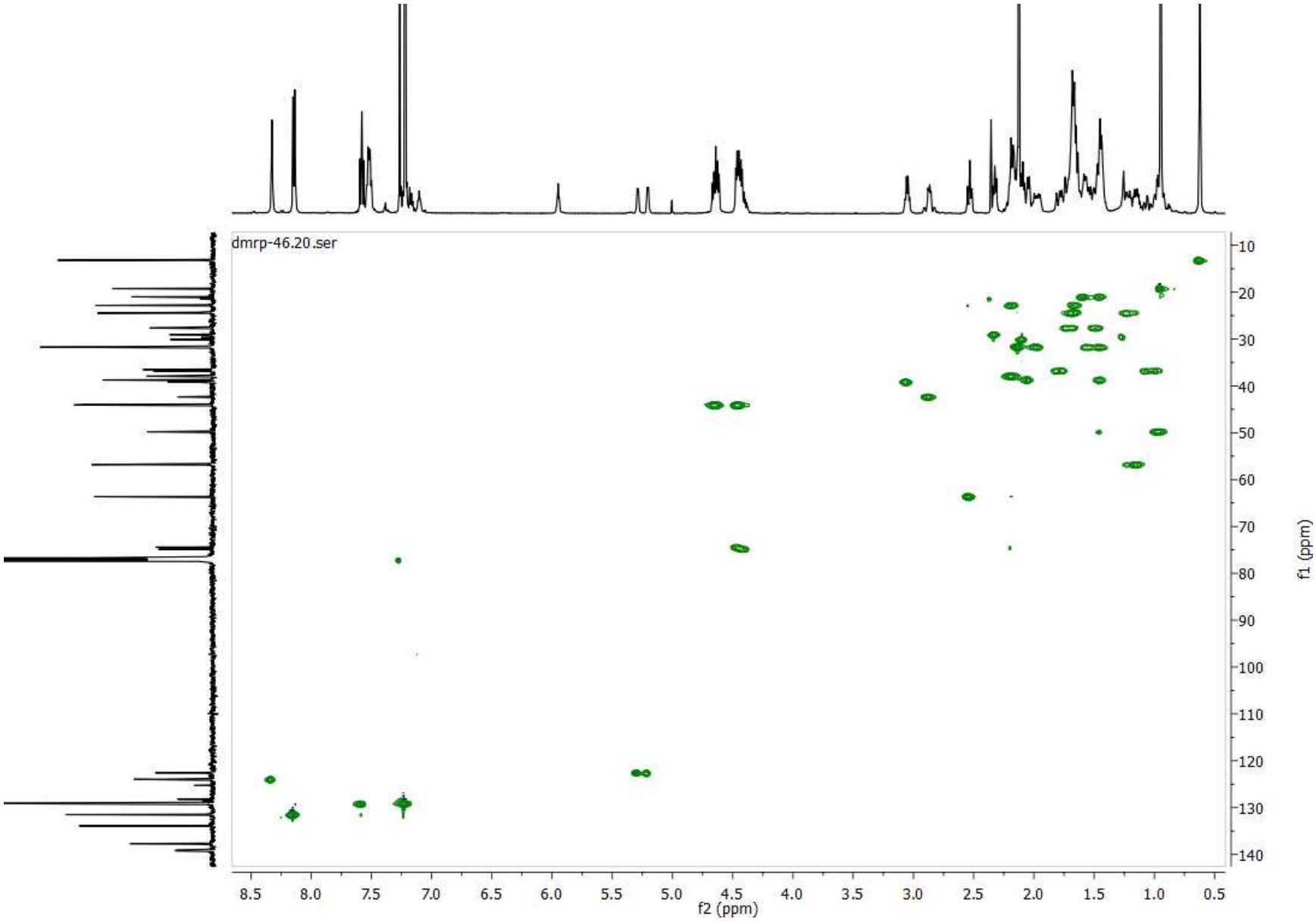
^{13}C NMR spectrum of rotaxane **4b** in $CDCl_3$



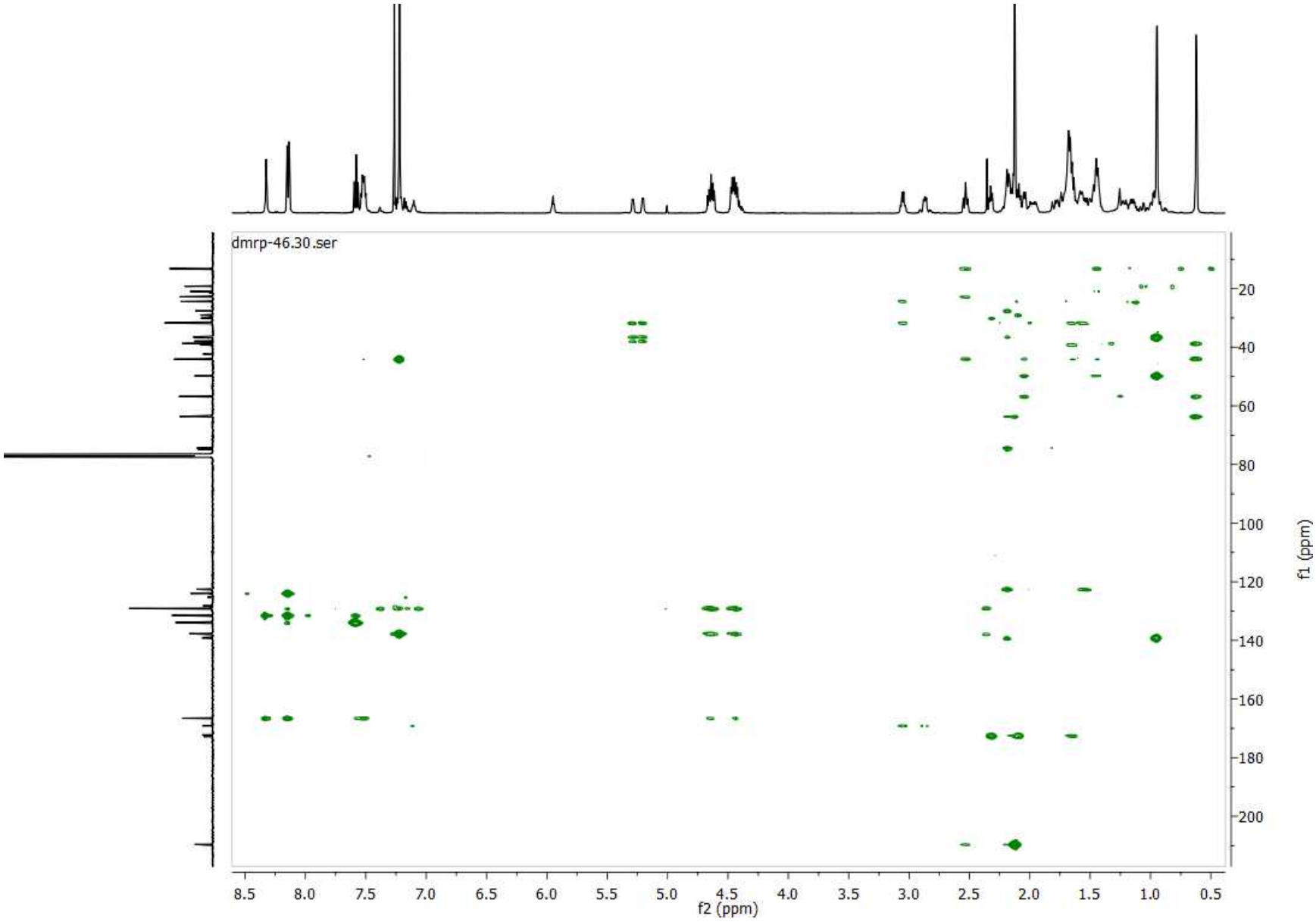
COSY spectrum of rotaxane **4b** in $CDCl_3$



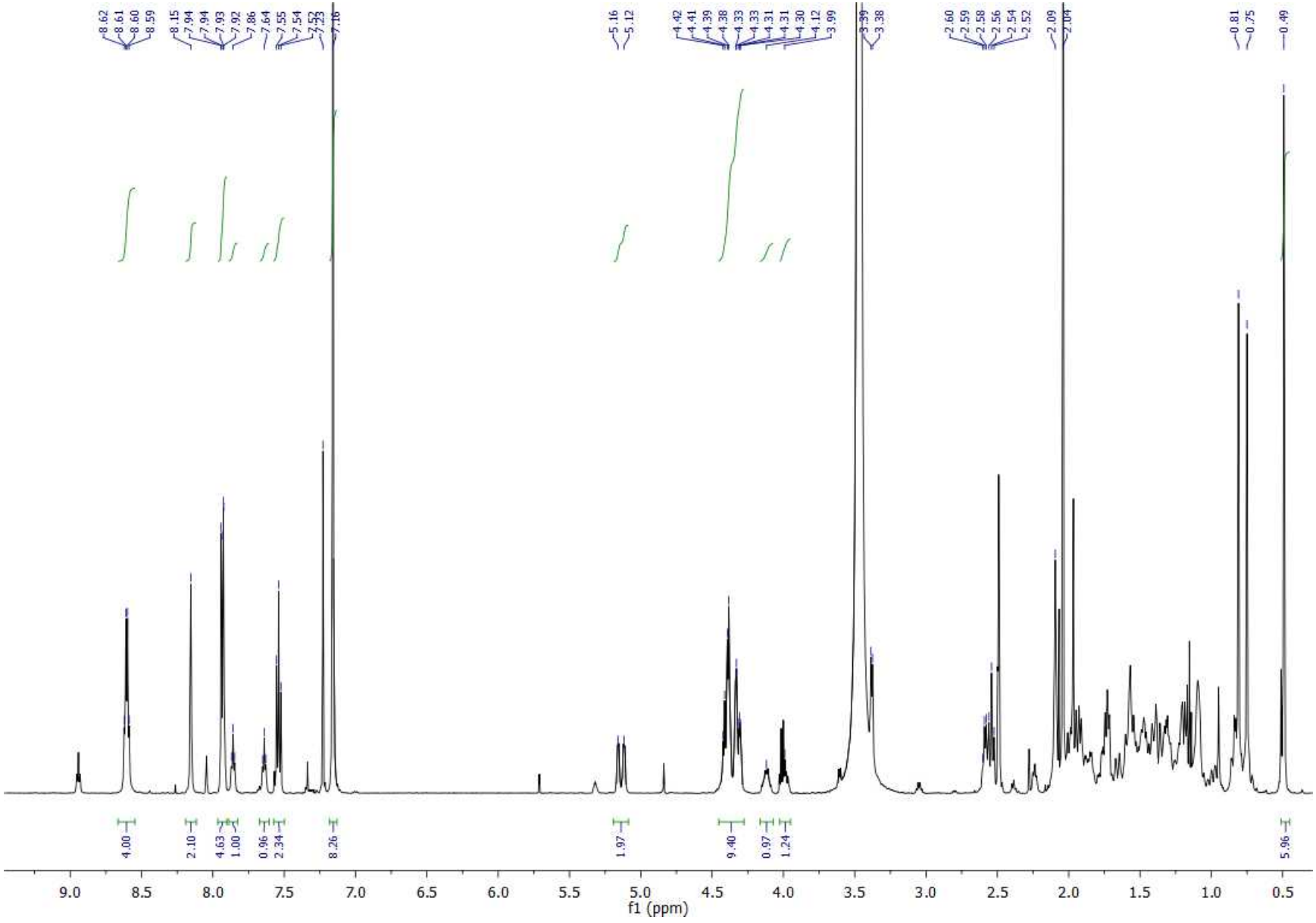
HSQC spectrum of rotaxane **4b** in $CDCl_3$



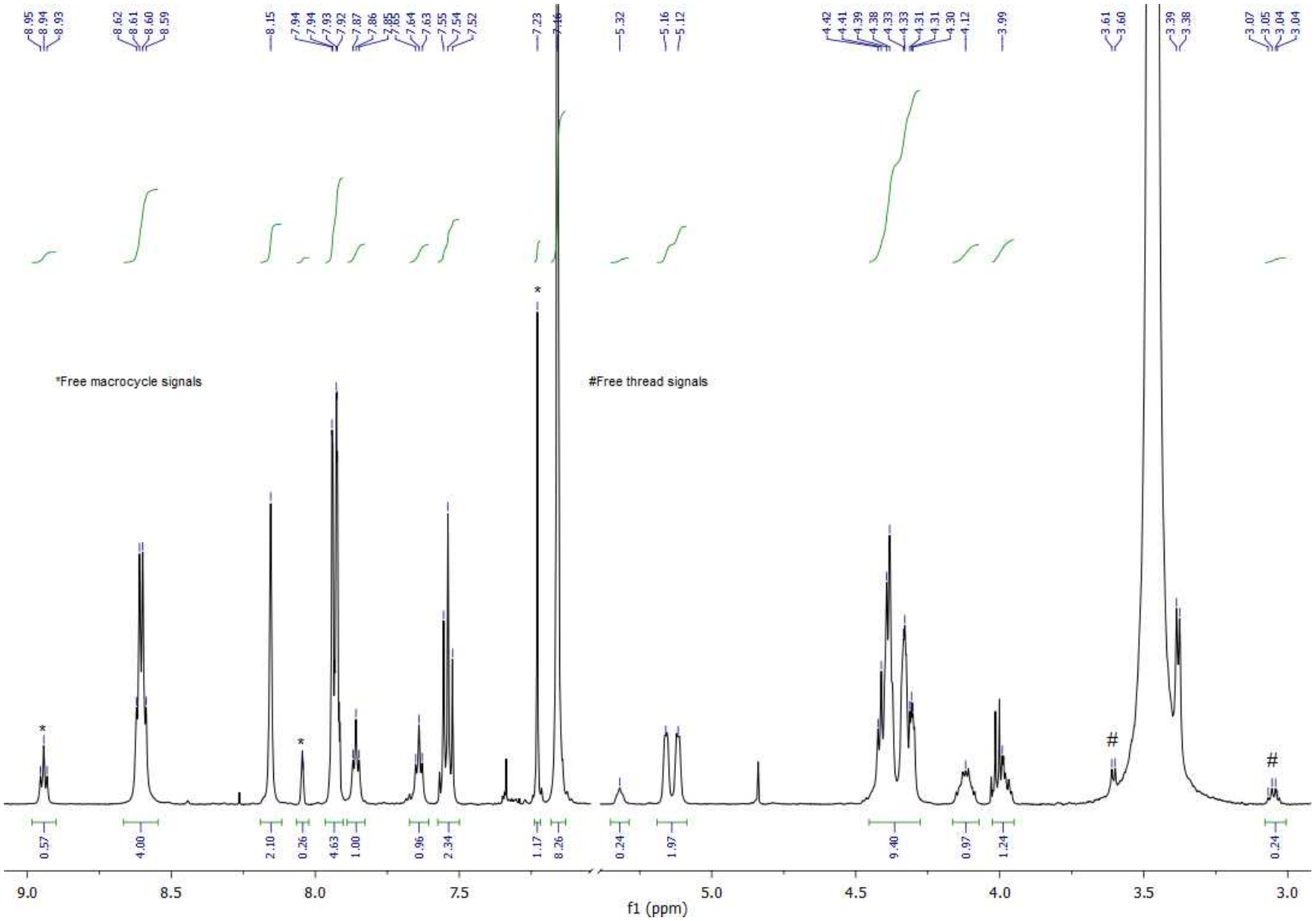
HMBC spectrum of rotaxane **4b** in $CDCl_3$



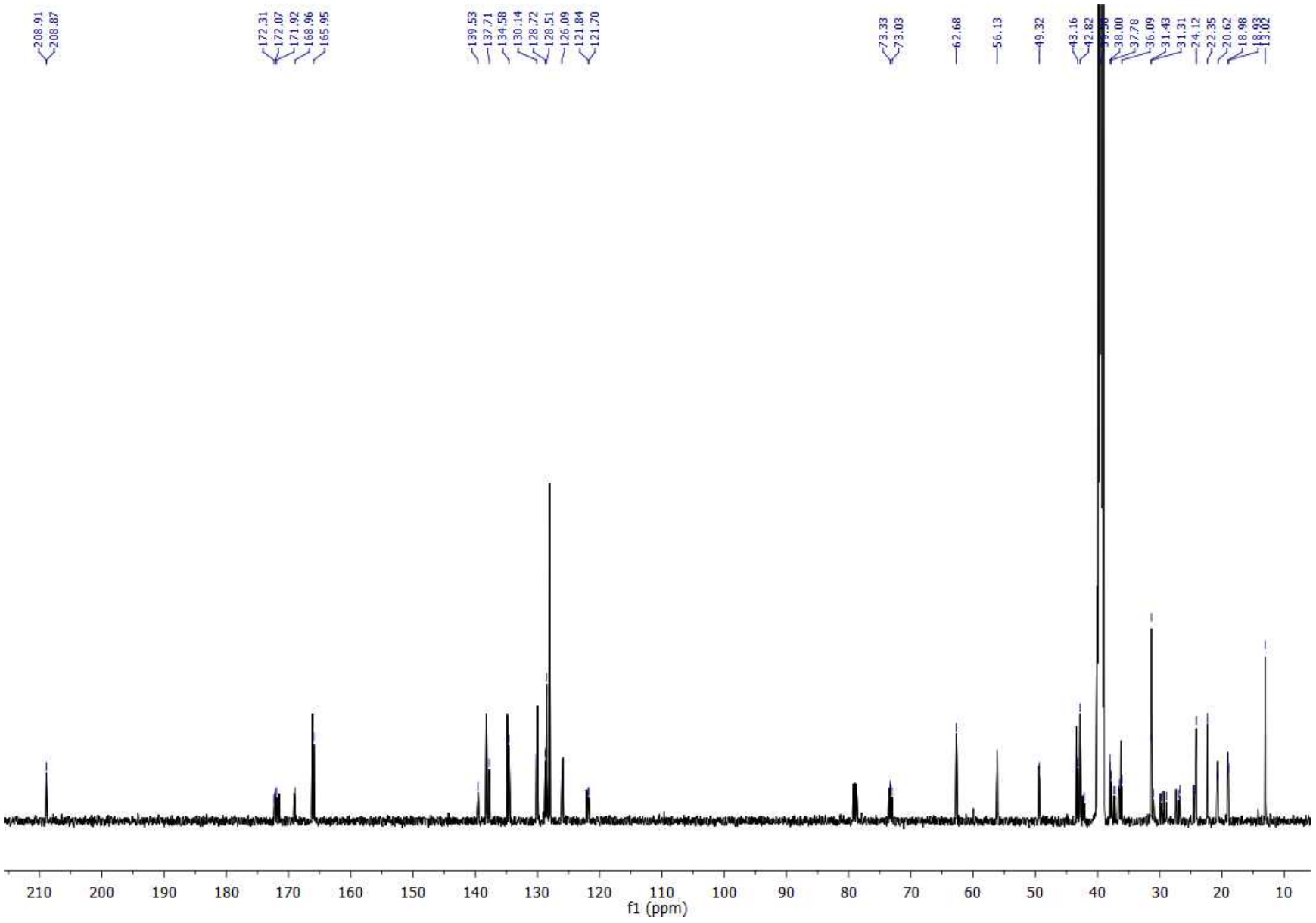
^{1}H NMR spectrum of rotaxane **4b** in [D_6]DMSO



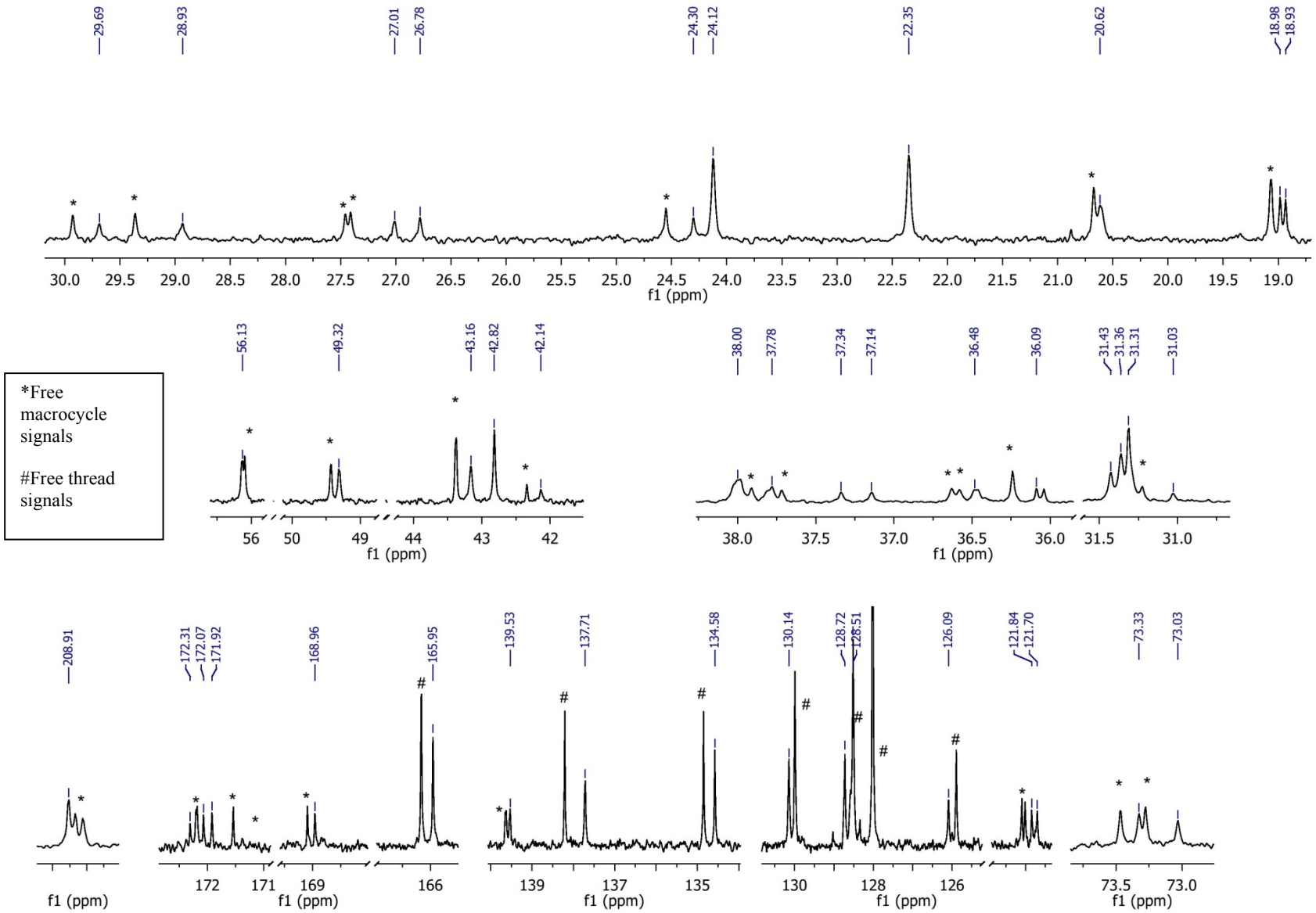
Expanded part of ^{1}H NMR spectrum of rotaxane **4b** in [D_6]DMSO



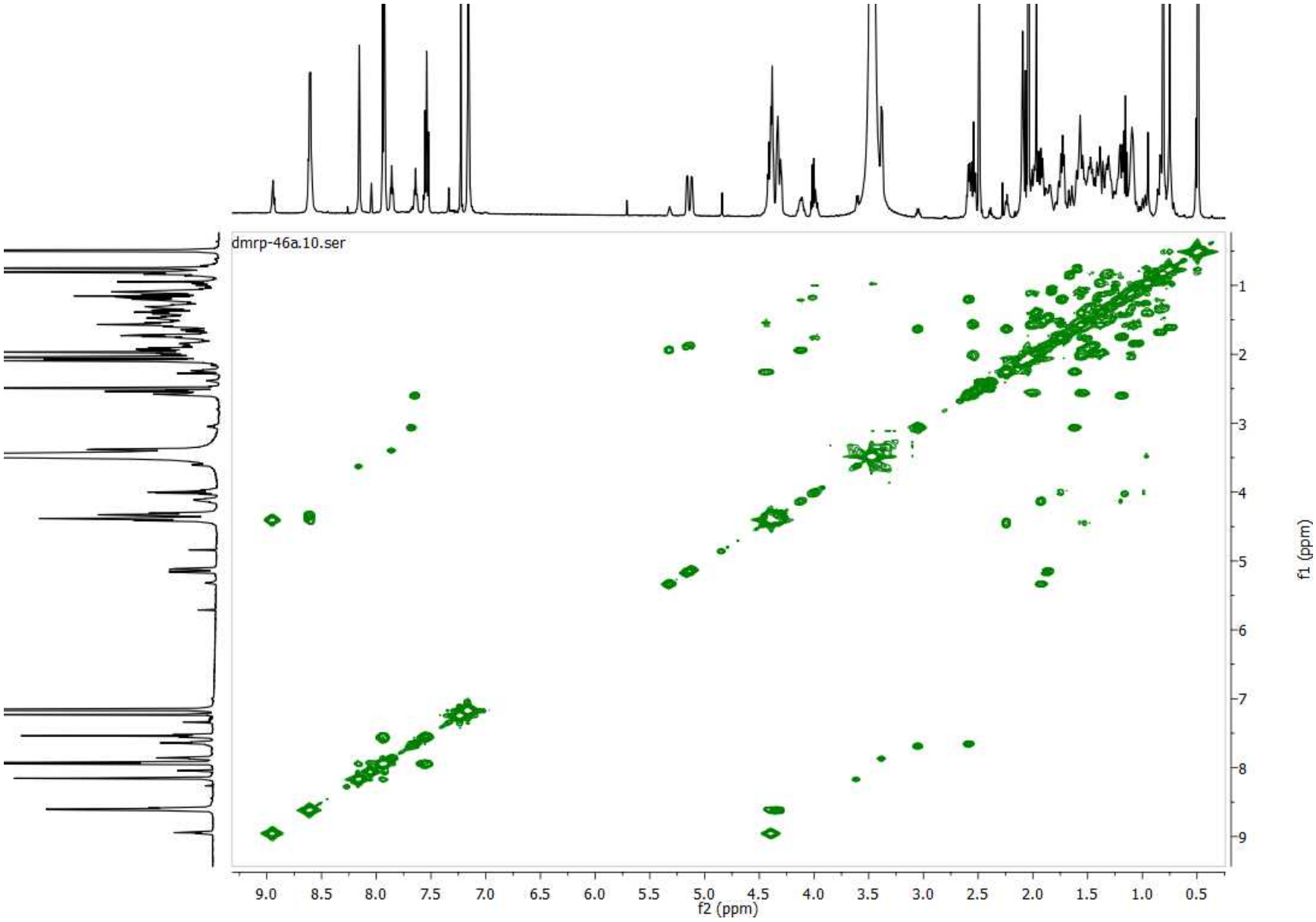
^{13}C NMR spectrum of rotaxane **4b** in [D_6]DMSO



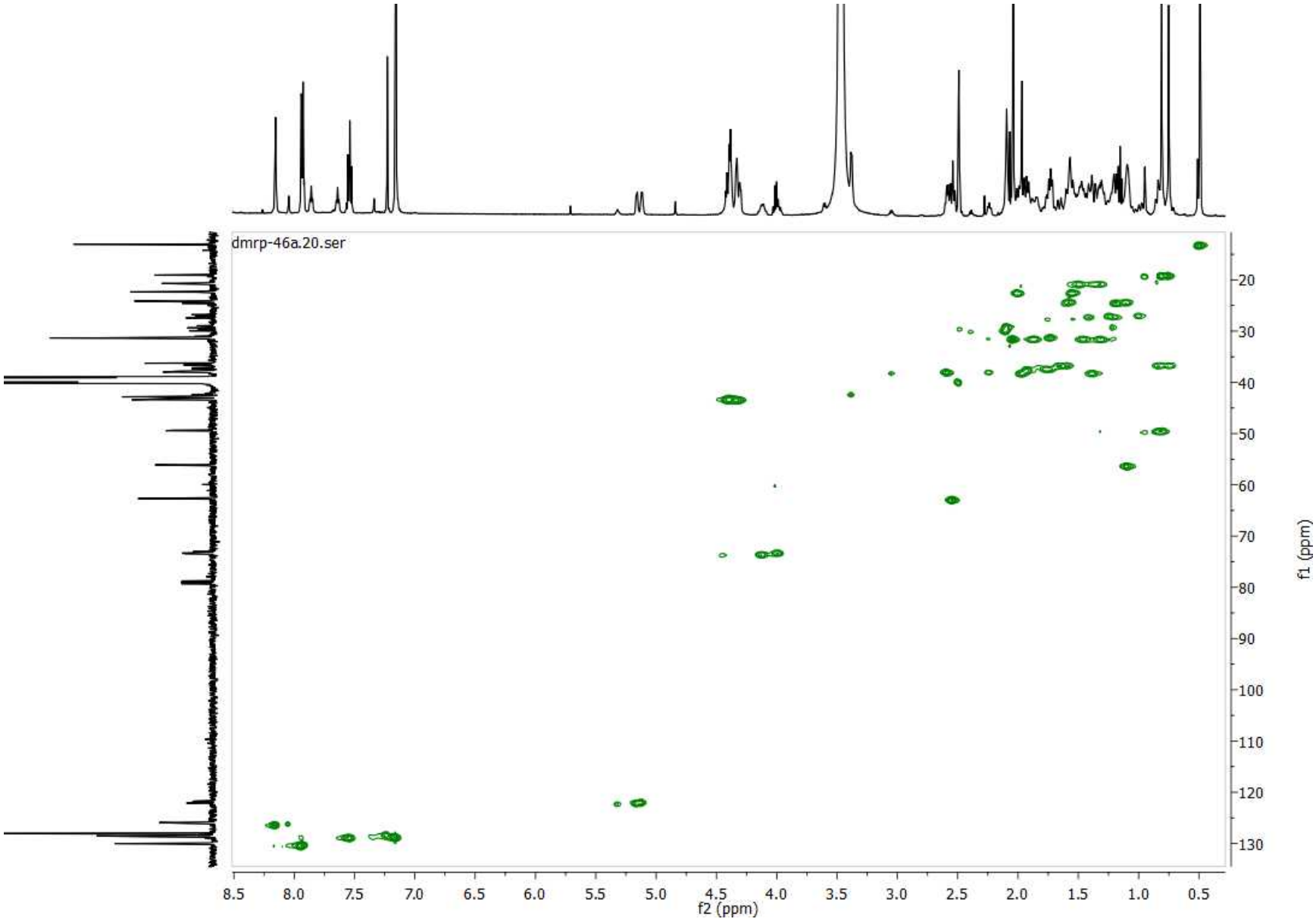
Expanded parts of ^{13}C NMR spectrum of rotaxane **4b** in [D_6]DMSO



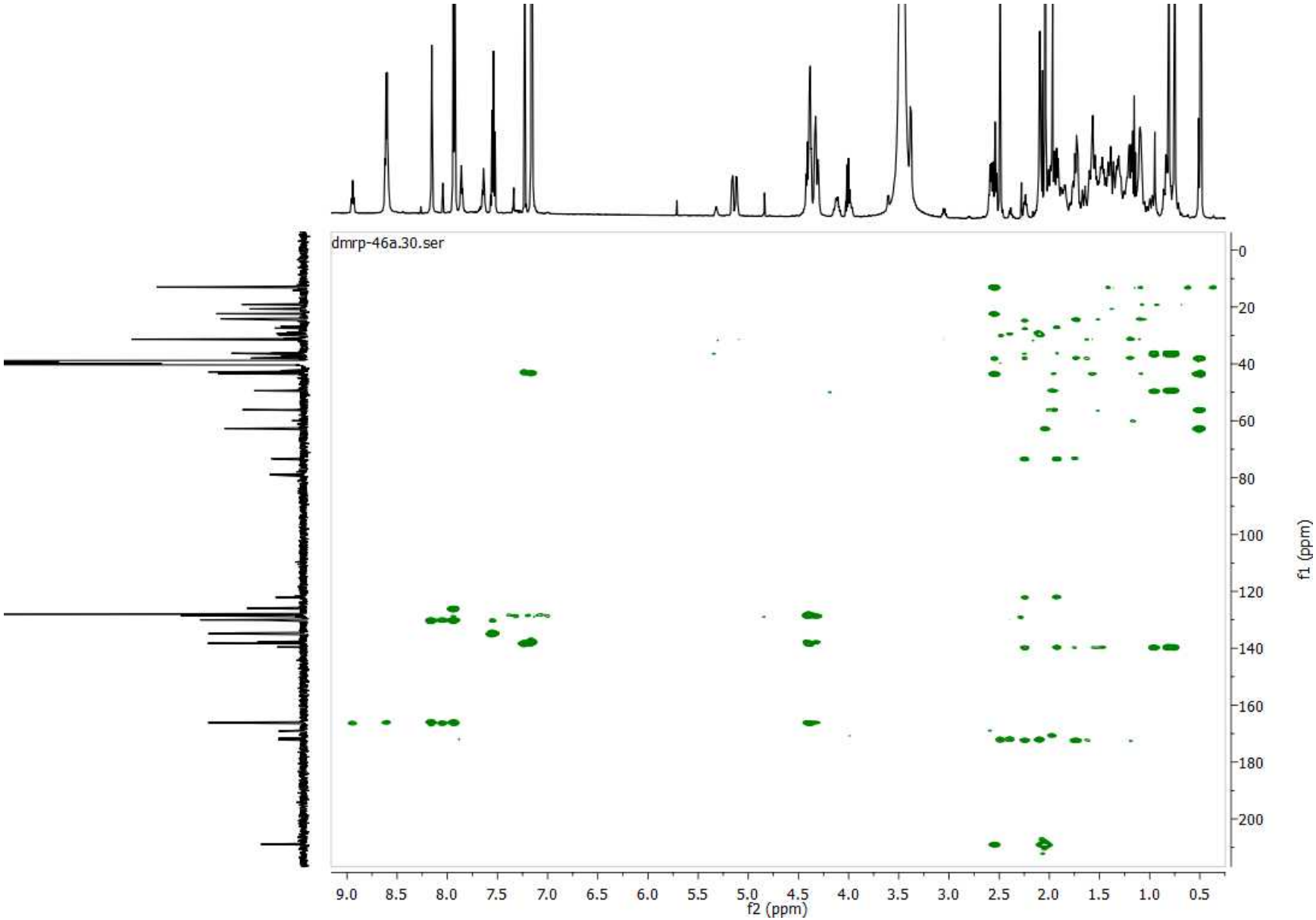
COSY spectrum of rotaxane **4b** in [D_6]DMSO



HSQC spectrum of rotaxane **4b** in [D_6]DMSO

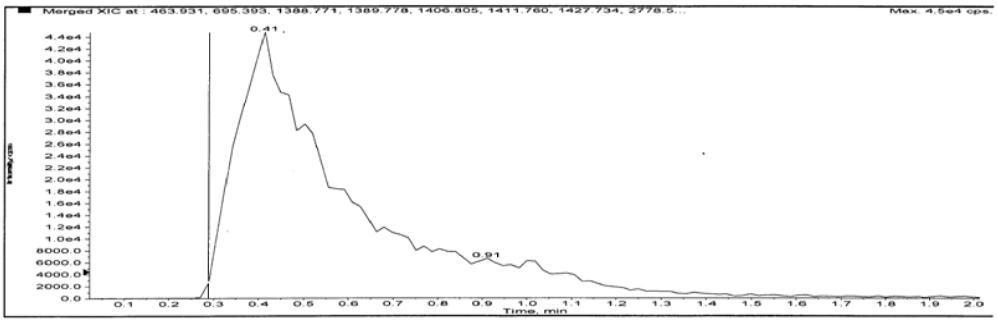


HMBC spectrum of rotaxane **4b** in [D_6]DMSO

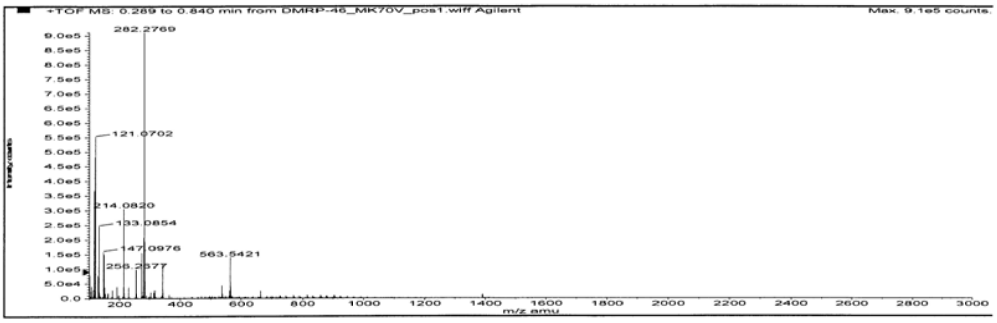


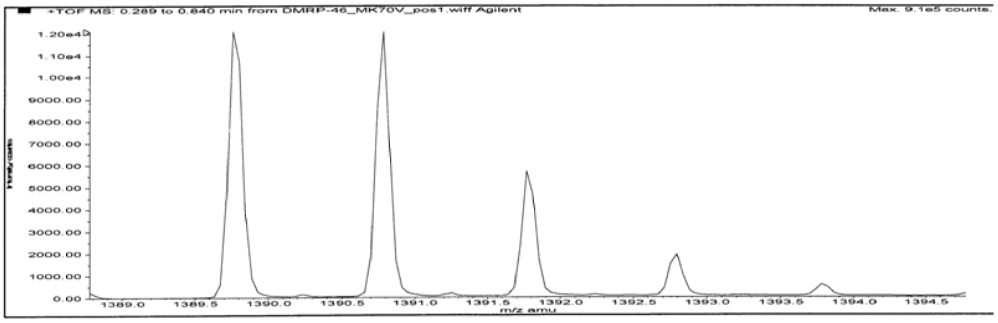
DMRP-46

Sample Name: **DMRP-46** Sample Location: **P1-E4** Sample Id: Operator: **Milka**
Data File Name: **D:\PE Sciex Data\Projects\D_Milic\Data\DMRP-46_MK70V_pos1.wiff** Acq Time: **July 18 2014, 01:53:03 PM**
Method: **d:\TOF_Data\damethods\Night_Seq_Comp_ident1.anm\efc.xml**



Merged XIC, Period# : 1 Experiment# : 1





Formula	Compound name	Mass	Peak RT (min)	Peak area	Description
C84H104N6O12	--	1388.77122	0.41	5.71494 E5	--

Species	Abundance (counts)	Ion Mass	Measured Mass	Error (mDa)	Error (ppm)	Ret. Time Error (min)
[M+H]+	12604.35	1389.77850	1389.77647	-2.03206	-1.46	--

Mass spectrum of rotaxane **4b**

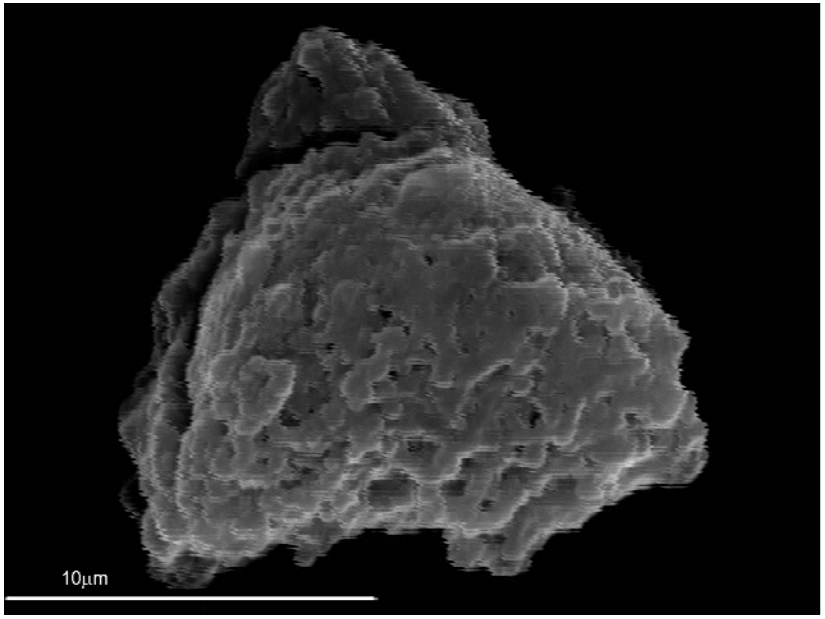


Figure S1. SEM image of thread **3b (0.1 mM in PhMe/EtOAc=1:1)**